ADSORPTION OF ARSENIC USING THE ACTIVATED CARBON PREPARED FROM COCONUT COIR

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CERTIFICATION OF APPROVAL

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by

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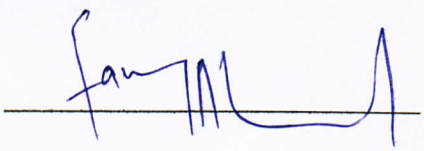

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or person.

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ABSTRACT

The presence of arsenic over the permissible concentration in environment can be unfavourable to living species. Coconut coir, an agriculture waste product has been treated chemically and thermally and used as adsorbent in this project. The adsorption characteristics of arsenic onto activated carbon prepared from coconut coir from aqueous solution has been investigated. The activated carbon prepared from coconut coir is referred as CC where else Merck activated carbon is referred as AC. The focus of the project is the potential of CC for the removal of arsenate.

The adsorption of arsenate is explored by varying the initial concentration of arsenate solution, pH of solution and the amount of adsorbent used at 27°C ± 2..Batch kinetic studies shows that an equilibrium time of 0.5h is required for the adsorption of arsenate on both AC and CC. The experimental assessment shows that adsorption of arsenate ions from aqueous solution is dependent on the initial concentration of solution, pH of solution and the amount of adsorbents.

With an increase in the concentration of arsenate solution from 5ppm until 40 ppm, the adsorption of arsenate increased dramatically. However, when the concentration is increased to 100 ppm, the adsorption of arsenate is decreased. The adsorption of arsenate is proven to be pH dependent. The maximum arsenate adsorption is found to be in the pH range of 5 and 7. At a very acidic (pH 2) and a very basic (pH 8) environment, the adsorption of arsenate is impeded. The maximum adsorption of arsenate is when the amount of adsorbent is 1.0 g. The adsorption of arsenate is hindered when the amount is multiplied to 2.0 g.

The adsorption of arsenate onto CC and AC respectively is quite comparable. Though, CC is economically viable compared to AC, thus CC has the potential to be explored as the future activated carbon for the removal of arsenic from aqueous solution via adsorption process.

ACKNOWLEDGEMENT

First and foremost, I would like to express my praises to God for His blessing.

My deepest appreciation and gratitude is extended to my supervisor, Dr. Farooq Ahmad for being very encouraging, supportive and responsive throughout the whole process of completing this final year project to fulfil the university requirement. Without his constant supervision and guidance, I may not be able to complete this project successfully.

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TABLE OF CONTENTS

ABBREVIATIONS AND NOMENCLATURES

A.A.S	Atomic Absorption Spectrometry
AC	Commercialized Activated Carbon
As(V)	Arsenate ion
CC	Coconut Coir Activated Carbon
EDX	Energy dispersive X-ray spectroscopy
FBAU	Fixed Bed Activation Unit
HCl	Hydrochloric Acid
KOH	Potassium Hydroxide
NaOH	Sodium Hydroxide
Na_2CO_3	Sodium Bicarbonate
Na_3AsO_4	Sodium Arsenate
ppm	parts per billion
SEM	Scanning Electron Microscope
TGA	Thermogravimetric Analysis
VDW	Van der Waals
XRD	X-ray diffraction

CHAPTER 1
INTRODUCTION

1.1 Background of Study

The presence of arsenic over the permissible concentration in environment can be unfavorable to living species. In most aquatic system, the elimination of arsenic from aqueous solution especially drinking water is extremely important to the public health.

Many methods have been proposed for arsenic removal such as coagulation, chemical precipitation, electro-analysis, ion exchange and adsorption. Among these processes, adsorption process via appropriate adsorbent can be an effective technique for the removal of arsenic from aqueous solution [1].

The common adsorbent in industry is activated carbons (AC). AC has the potential as a solid adsorbent for the removal of heavy metal namely arsenic from aqueous solution via adsorption process. AC has been proven to be effective for the removal of arsenic [2].

Increasing concern over pollution of water arising from different industrial activities promotes growing demand of AC. Despite the expensive AC, for efficient removal treatment via adsorption, vast quantities of AC are required. Hereby, the expenses of adsorption are slightly overpriced. Therefore, a cheaper alternative is necessary for the betterment of the society and people.

The potential alternative for the existence AC has been intensively investigated [3]. The substitute for AC should be effortlessly available and cheaper. Recent research has focused on the development of unconventional low-cost adsorbents using the various agricultural, industrial, natural or biological wastes materials.

Coconut coir, being one of the many abundant agriculture waste materials available in Asia especially Malaysia, makes a perfect potential agent that acts as AC in the adsorption process for the removal of arsenic in aqueous solution. The annual production of coconut coir is generated from the coconut fruit during the coconut coir fiber preparation process for mattress padding industry [4].

The present study is initiated to evaluate the effectiveness of AC prepared from coconut coir as an adsorbent in the adsorption process for the removal of arsenic from aqueous solution. Laboratory batch adsorption and adsorption kinetic studies are conducted to determine the adsorption capacity of coconut coir AC.

The parameters that may affect the adsorption process are the initial concentration of adsorbate, pH of solution and the amount of adsorbent. The equilibrium time is evaluated based on the contact time. The adsorption capacity of activated carbon prepared from coconut coir is compared to commercialized activated carbon

1.2 Problem Statement

1.2.1 Problem Identification

Arsenic contamination of natural resources has emerged as one of the major public health problems in many countries. Over eighty percentages (80%) of arsenic produced by humankind is used in environmentally dissipative manners namely, herbicides, insecticides, wood treatments, chemical agents and drugs. The pollution of arsenic in aqueous solution has become severe with the development of economy.

Many cases of arsenic poisoning in drinking water have been reported in Taiwan, China, India, Bangladesh, Thailand, Chile, Argentina, Mexico, Canada and the United States of America. About fifty million people in Bangladesh [5] and six million in China are estimated at risk of being poisoned by ingesting water with arsenic levels above 0.05 mg/L. Overall worldwide at-risk population is estimated to be over hundred-fifty million.

The wastewater from the industries such as metallurgy, mining, chemical pharmacy, chemical plant, pesticide product and leather tanning often include the arsenic pollution [6]. Furthermore, a large percentage of the arsenic from industrial wastes is not treated, instead blindly released directly into groundwater, namely areas around gold mines, municipal dump sites, power plants that burn arsenic-rich coal and base metal smelters [7].

Recent epidemiological studies validate the fact that the exposure to inorganic arsenic in drinking water increases the risk of skin cancer and other internal tumors of bladder, liver, kidney and lung [8]. Chronic exposure to low levels of arsenic can affect the skin, circulatory system, gastrointestinal tract, nervous system and heart. Reproductive effects such as malformations, low birth weight and spontaneous abortion have been increasingly reported due to long term exposure to arsenic [9].

According to Pauline L. Smedley (2003), World Health Organization (WHO) has had a public position on arsenic in drinking water since 1958. The last edition of WHO Guidelines for Drinking Water Quality 2008 (GDWQ) established 0.01mg/L as provisional guideline value arsenic in drinking water for health protection [7].

1.2.2 Significance of Study

Adsorption process has been comprehensively successful method for heavy metal ions removal at low concentration from aqueous solution [10]. Adsorption process is viable because it effectively removes heavy metal namely lead, copper and arsenic. Moreover, according to B. Bayat (2006), it is significantly economical compared to other removal process [11]. In principle, adsorption is universal, inexpensive and rapid.

Despite the effective adsorption process, the bulk amount of AC needed is slightly costly. Therefore, AC may not be cost-effective for large volumes of water treatment especially for drinking water. Hence, it is vital to explore the potential of low cost adsorbents developed from waste biomass, industrial waste, mineral wastes and agriculture wastes [12].

Quoting Yogesh C. Sharma, Uma and Siddh N. Upadhyay, (2009), coconut coir has been discovered as an alternative AC for the adsorption process for the removal of arsenic from aqueous solution. Being resistant to biodegradation, it is heaped along road sides [12]. Since the material is rich in cellulose content, an effective solution in the use of the material for the preparation of AC.

Hence, a feasibility study is carried out in order to exploit the potential of coconut coir as adsorbent for removal of arsenic from aqueous solution via batch adsorption process.

1.3 Objective of Project

The primary objective of this project is to perform an experimental comparative assessment to elucidate the effectiveness of activated carbon prepared from coconut coir on the removal of heavy metal namely arsenic via batch adsorption process.

The secondary objective is to discover the optimum parameters to maximize the adsorption process. The three parameters explored are the initial concentration of arsenic solution, pH of solution and the amount of adsorbent used.

The third objective is to compare the adsorption capacity using the conventional activated carbon and the activated carbon prepared from coconut coir

1.4 Scope of Study

The overall adsorption study is to investigate the ability of activated carbon prepared from coconut coir to remove arsenic from aqueous solution over a range of operating conditions. The project gives more emphasis on arsenate which is the most prevalent form of arsenic in drinking water sources.

The performance of coconut coir as an adsorbent for removal of arsenic from aqueous solution via adsorption is explored by running the batch adsorption experiments. The parameters investigated include the initial concentration of solution, pH of solution and the amount of adsorbents.

Kinetics tests are conducted in order to determine the equilibrium contact times. Then, the batch adsorption tests are conducted to establish the conditions that are favourable for arsenic removal.

A comparison study is also conducted to determine the efficiency of the activated carbon prepared from coconut coir and the commercialized activated carbon.

CHAPTER 2
LITERATURE REVIEW

2.1 Arsenic Chemistry and Occurrence in Water

Arsenic is a metalloid that occurs in a variety of forms and oxidization states and valences [8]. According to Alan H. Wech and Kenneth G. Stollenwerk (2003), under typical pH condition, As(V) exists as an anion. In contrast, As(III) is fully protonated (positively charged) and exists as uncharged molecules [2]. About 25 percentage of all community water suppliers worldwide would violate an arsenic standard of 0.01 mg/L arsenic are exposed to long term risk of arsenic [9].

Arsenic is a common pollution in industries by human activities and natural phenomena. It has long been recognized that the smelting of non-ferrous metals and the production of energy from fossil fuel are the two major industrial processes that lead to arsenic contamination of water [14].

Arsenic cannot be destroyed in the environment. It can only change its form, or become attached to or separated from particles [8]. It may change its form by reacting with oxygen or other molecules present in air, water, or soil, or by the action of bacteria that live in soil or sediment.

Arsenic released from power plants and other combustion processes is usually attached to very small particles. It pollutes the lakes, rivers, or underground water by dissolving in rain or snow or through the discharge of industrial wastes [8]. Some of the arsenic will stick to particles in the water or sediment on the bottom of lakes or rivers, and some will be carried along by the water.

Long term exposure to arsenic in drinking water has been implicated in a variety health concern including several types of cancer, cardiovascular diseases, diabetes and neurological effects. Soluble inorganic arsenic is acutely toxic, and ingestion of large doses leads to gastrointestinal symptoms, disturbances of cardiovascular and nervous system functions, and eventually death [9].

According to William T.Frankenberger, Jr, (2002) [14],

> The dissolved forms of arsenic in seawater contain arsenate; As (III), arsenite; As (V), monomethylarsonic acid (MMA) and dimethylarsinic acid (DMA). As (III) is the major arsenic found in drinking water whereas arsenite occurs as significant constituent. Arsenate is the predominant species but the arsenite is considered to be more hazardous. (p.97)

2.2 Potential Acute Health Effects

While conducting experiment in laboratory, it is essential to identify the potential of arsenic exposure risk. There are four possible acute health effects including inhalation, ingestion, skin contact and eye contact. Via inhalation, arsenic may cause inflammation of the mucous membranes with cough and restlessness. Symptoms are due to inhalation and ingestion that may cause pulmonary edema.

Referring to F.W. Pontius, K.G. Brown, C,-J. Chen (2007), arsenic is highly toxic [9]. If ingestion, it may cause burning in esophagus, vomiting and blood diarrhea. Symptoms of cold and clammy skin, low blood pressure, weakness, headache, cramps, convulsion and coma may follow. It may cause damage to liver and kidney.

Furthermore, while handling arsenic, skin and eye contact should be avoided. Through skin contact, it may cause irritation including redness, itching and pain where else, through eye contact, itching, burning, watery eyes and damage of conjunctiva.

Therefore, complete personal protection is necessary to prevent health hazards. While managing the arsenate solution, it is crucial to work in the fume chamber. Besides, splash goggles, lab coat, dust respirator and impervious gloves should be intact to avoid haphazard accident.

2.3 Fundamentals of Adsorption Processes

2.3.1 Theory

Adsorption refers to the accumulation of a substance at the interface between two phases such as the liquid and solid [15]. The substance that accumulates at the interface (adsorbs) is referred to as the adsorbate and the non-aqueous phase on which the adsorption occurs is called adsorbent.

As cited by Parinda Suksabye, Paitip Thiravetyan and Woranan Nakbanpote (2007), adsorption is the accumulation of a substance in the interior of the non-aqueous phase [15]. Adsorption tests may be conducted using either batch or continuous flow reactors such as packed bed column. Typically, complete mix reactors are used to develop adsorption isotherm relationship where the profile of the adsorbate concentration in the effluent as a function of time.

Figure 1 shows the adsorption process in bulk adsorbate fluid phase. In aqueous solution, the charges of adsorbate define the efficiency of the adsorption process. Due to the surface charges of the adsorbate and pores on the adsorbent (activated carbon), adsorbates are driven to occupy the surface pores. Therefore, the removal of adsorbate from the aqueous solution is said to be successful

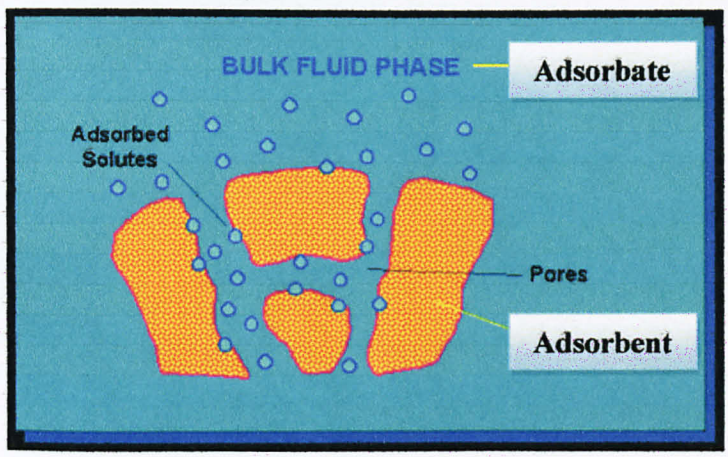


Figure 1 : Illustration of adsorption process

As cited by B. Amaransinghe and R.A. Williams (2007), adsorption is due to from Van der Waals interactions (physical adsorption) or it can have the character of a chemical process (chemical adsorption)[17].

For an efficient adsorption process, the criterion of adsorbent plays a big part. The followings are the selection criterion for adsorbent [16];

i. High selectivity to enable sharp separations
ii. High capacity to minimize amount adsorbent needed
iii. Favorable kinetic and transport properties for rapid sorption
iv. Chemical and thermal stability to preserve amount and its properties
v. Hardness and mechanical strength
vi. High fouling resistance
vii. Capability of being regenerated - relatively low cost

2.3.2 Development of Adsorption Isotherms

Adsorption isotherms are developed by exposing a given amount of adsorbate in a fixed volume of liquid to various amount of activated carbon. According to P. Roonasi [42], to develop adsorption isotherms, the equation 1 stated below is used.

$$q_e = \frac{(C_0 - C_e)V}{m} \qquad \text{.....Equation 1}$$

where,

q_e = Adsorbent phase concentration after equilibrium, (mg adsorbate/g adsorbent)

C_0 = Initial concentration of adsorbate, (mg/L)

C_e = Final equilibrium concentration of adsorbate after adsorption, (mg/L)

V = Volume of liquid, (L)

m = mass of adsorbent, (g)

2.4 Activated Carbons (AC)

AC has been widely used in environmental pollution control as an adsorbent for adsorption process for removal of heavy metal from aqueous solution (refer to Figure 2). Without any surface treatment, activated carbon provides zero adsorption capacity for the metal ions. They have proved to be effective for the removal of wide variety of pollutants from aquatic system.

Based on Emirbas, Kobya, Senturk and Ozkan (2004), adsorption on AC has been recommended to accomplish the permissible limits for toxic metals and organic pollutants [16],. Nevertheless, adsorption process is economically inappropriate due to vast quantities of costly AC is required for full scale water treatment.

Therefore, the utilization of agricultural waste materials is increasingly becoming a vital concern because these wastes represent unused resources and in many cases present serious disposal problems.

Quoting Amaransinghe and Williams (2007) [17],

> Numerous waste biomass sources are available which some experimental adsorption has been reported, namely rice husk, coconut shell, saw dust, dry tree leaves and barks and tea and coffee waste. Plant materials are mainly comprised of cellulose materials that can adsorb heavy metal ions in aqueous solution (p.300).

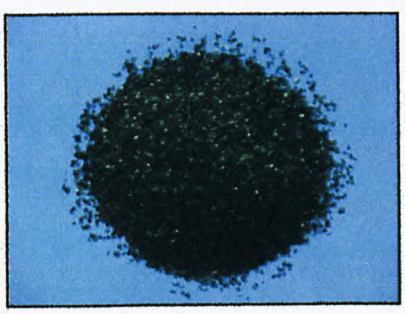


Figure 2 : Commercialized Activated Carbon Merck (2mm)

2.5 Coconut Coir

Coconut is a single seed of a fruit of a coconut palm tree called Cocos Nucifera (refer to Figure 3). As mentioned by P.Suksabye and P.Thiravetyan (2007), although coconut palms grow throughout the world's tropical regions, the vast majority of the commercially coir comes from India and Sri Lanka [18].

Coconut coir pith is an agriculture waste from coir industry. It is renewable resource where it can be treated to be an activated carbon for adsorption process [4]. The elastic cellular cork pithy material forms the non fibrous tissues of the coconut husk. One ton of coir pith is produced for every 10 000 husks used in the coir industry.

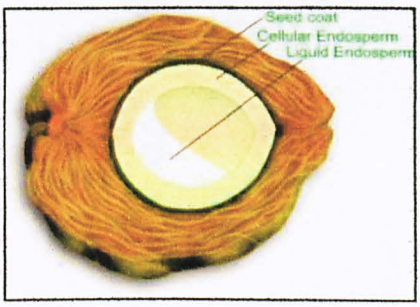


Figure 3 : Interior layer of a coconut fruit

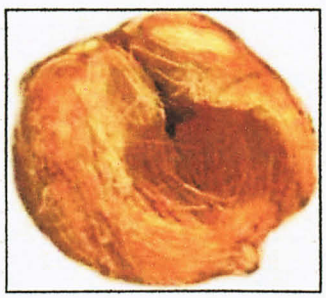


Figure 4 : Coconut Coir Fiber

In India, only 15% of the husk fibers (refer to Figure 4) are actually recovered for use where the total production is about ¼ of the world's 55 billion coconut each year.

According to C Namasivayam (2003) [19],

> Agricultural wastes like coir pith are discarded in the agricultural sector in Malaysia. Annual production is around 7.5million ton. Coir pith which constitutes as much as 70% of the coconut husk is light fluffy material that is generated in the separation process of the fiber from the coconut husk. Coir pith is discarded as a waste and its accumulation around coir processing centers has been a menace.

The accumulation of coir pith near coir factories in large quantities causes solid waste pollution problem. Since it is composed of lignocellulosic compounds, total degration of these compounds is considered an impossible task.

Figure 5 : The abundance coconut coir waste heaped by the roadside

The individual fiber cells are narrow and hollow with the thick walls made of cellulose (refer to Table 1). ACs prepared from coconut coir has been successfully used in removal of dyes, 2-chrolophenol, and heavy metal [20]. Coconut coir is biodegradable but takes 20 years to decompose.

According to Parinda Sukbaye (2008) [4],

Table 1 : Coconut Coir Compound

Coconut Coir compound (dry basis)	
Compound	**Percent (%)**
Cellulose	33.61
Lignin	36.51
Pentosans	29.27
Ash	0.61

2.6 Characterization of Coconut Coir

2.6.1 Energy Dispersive X-ray Spectroscopy

Energy dispersive X-ray spectroscopy (EDX) is used to identify the elemental composition of as little as a cubic micron of material. EDX works by detecting X-rays that will be produced by a sample placed in an electron beam. The electron beam excites the atoms in the sample that later produce X-rays to release the excess energy [21].

The energy of the X-rays is characteristic of the atoms that produced them, forming peaks in the spectrum. Individual elements can have more than one peak related to them and some peaks from different elements may overlap to a certain degree [22]. EDX is often attached to the Scanning Electron Microscope (SEM).

2.6.2 Scanning Electron Microscope (SEM)

Conventional light microscopes use a series of glass lenses to bend light waves and create a magnified image. SEM creates the magnified images by using electrons instead of light waves [23]. The SEM shows 3-dimensional images at much higher magnifications. The images are very detailed.

The images created without light waves are rendered black and white [24].It can be used to estimate the pore size of the activated carbon quantitatively. Pore entrances, clearly visible as dark irregular shapes on lighter membrane background.

2.6.3 X-ray Diffraction (XRD)

X-ray diffraction (XRD) is used to reveal the chemical structural changes associated with the sorption process [25].

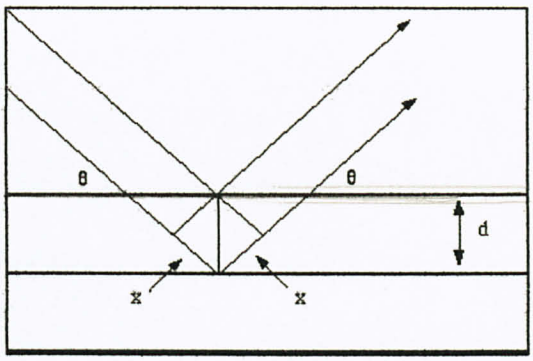


Figure 6 : Reflection of x-rays from two planes of atoms in a solid

2.6.4 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a tool that recognizes types of functional groups in a molecule [26]. It is by producing an infrared absorption spectrum that is like a molecular fingerprint. FTIR is used to identify chemicals that are either organic or inorganic.

By interpreting the infrared absorption spectrum, the functional groups in a molecule can be determined. FTIR spectra of pure compounds are so unique that they are called a molecular "fingerprint". Organic compounds have very rich, detailed spectra where else inorganic compounds are usually much simpler [27]

Bonds and groups of bonds vibrate at specific frequencies. A molecule that is exposed to infrared rays absorbs infrared energy at frequencies which are specific to that molecule. FTIR spectroscopy does not require a vacuum condition since neither oxygen nor nitrogen can absorb infrared rays. FTIR analysis can be applied to small quantities of materials, whether solid, liquid, or gaseous [28].

2.6.5 Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) used to measure the quantity and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere [29]. Measurements are used to determine the composition of materials and to predict their thermal stability at temperatures up to 1000°C.

The technique can characterize materials that demonstrate weight loss or gain due to decomposition, oxidation, or dehydration. Thermal stability of materials, oxidative stability of materials, composition of multi-component system, decomposition kinetics of materials, effects of reactive or corrosive atmospheres on materials, moisture and volatility can be found by using TGA [30].

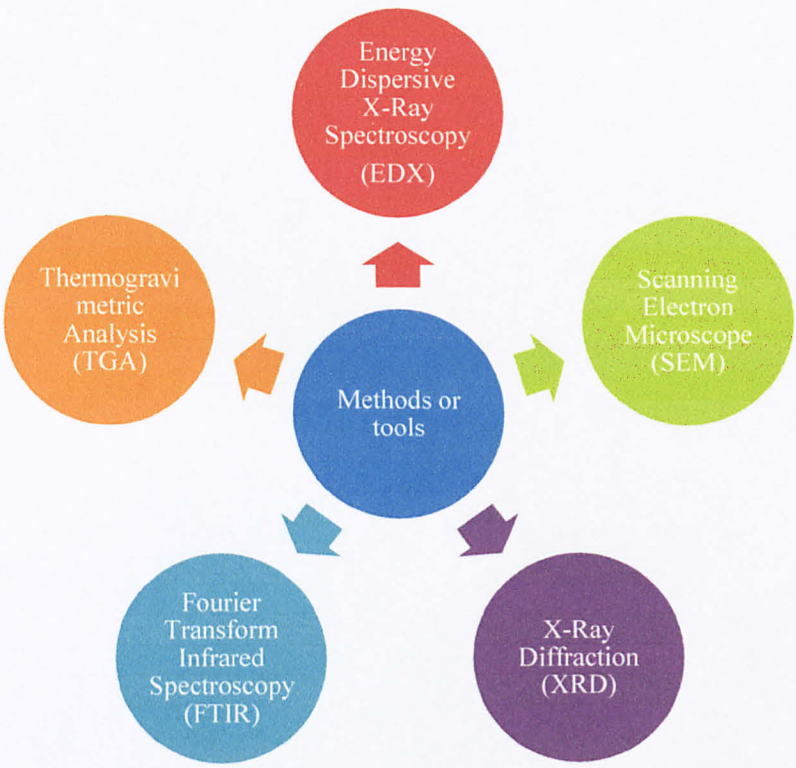
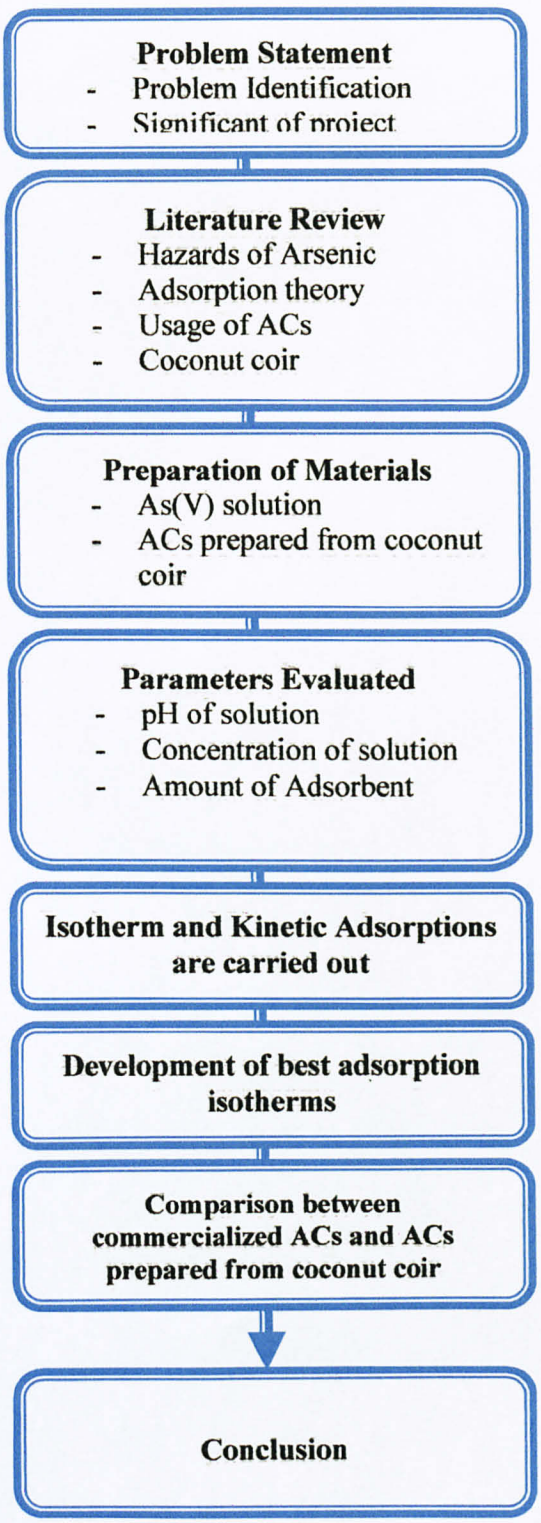


Figure 7 : Characterization of Treated Coconut Coir

CHAPTER 3

METHODOLOGY

3.1 Research Flow Chart



3.2 Project Activities

3.2.1 Flow of the Experiment

The batch studies have been conducted by mixing CC with arsenate solution prepared in the laboratory. The solution of arsenate is stirred at 120 rpm at varying time interval (0.5, 1.0, 1.5, 2.0, 3.0, 3.5 h) at 27°C ± 2 °C. A sample of 5mL is extracted to obtain the percentage of removal. The content is filtered through 0.45mm membrane filter using the vacuum pump and filtrate wash. Subsequently, it is analyzed for the remaining arsenate content using atomic absorption spectrophotometry (A.A.S.) at wavelength 213.9 nm.

Hereby the parameters are varied according to acquire the effects of pH, effects of initial arsenate concentration and effects of amount of adsorbent used.

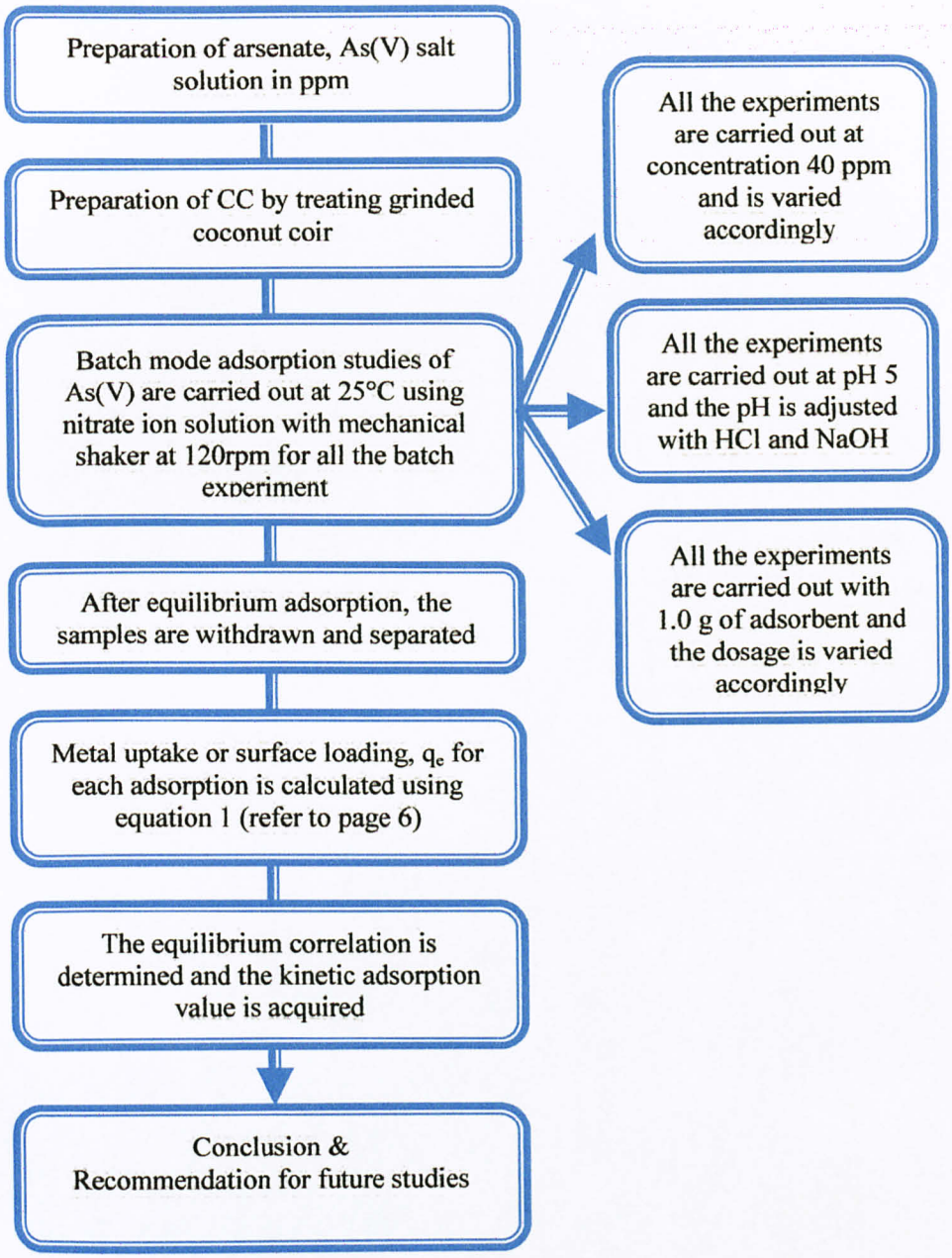
For the first parameter, initial arsenate concentration, the experiments are carried out at initial concentration of 5 ppm, 10 ppm, 40 ppm and 100 ppm at 27°C ± 2 °C. The pH of solution is set at pH 5 and the amount of adsorbent used is 1.0 g. It is assumed that the maximum adsorption process is at pH 5 using 1.0 g of adsorbent.

For the second parameter, pH of solution, the experiments are carried out at pH 2, pH 5, pH 7 and pH 8 at 27°C ± 2 °C. The pH is adjusted accordingly using NaOH and KCl. The initial concentration is set at 40 ppm and the amount of adsorbent used is 1.0 g.

For the third parameter, amount of adsorbent used, the experiments use 0.2 g, 0.8 g, 1.0 g and 2.0 g at 27°C ± 2 °C. The initial concentration is set at 40 ppm and the pH is set at 5.

Lastly, the effectiveness of the activated carbon prepared from coconut coir is compared to the conventional AC. The conventional AC used in this experiment is the AC from Germany named Merck. To avoid confusion, starting from this section, Merck AC is referred as AC where else the activated carbon prepared from coconut coir is referred as CC.

3.2.2 Overview



3.2.3 Preparation of Activated Carbon from Coconut Coir (CC)

1. Coconut coir is purchased from the nursery and is grinded for higher surface area. The grinded coconut coir is sieved to remove foreign components.
2. After the grinding and sieving steps, coconut coir is rinsed with distilled water. The rinsed coconut coir is later dried in oven for 24 hours at 105°C.
3. The weight of the coconut coir is evaluated to ensure constant weight, indicating the coconut coir is fully dried, containing no moist.
4. The dried and grinded coconut coir is sieved up to 400-600μm to ensure higher surface area for better adsorption.
5. To begin the treatment process, the dried coconut coir is soaked in 50 mL of 10% KOH in beaker.
6. The coconut coir in KOH solution (in beaker) is dried in oven for 24 hours (fully dried) at 105°C in oven till constant weight.
7. The treated coconut coir is then rinsed with distilled water, followed with 2% solution of Na_2CO_3 to remove odor. The treated coconut coir is placed at room temperature (27°C) in 2% Na_2CO_3 solution overnight.
8. Afterwards, coconut coir is rinsed with distilled water and is placed in oven at 105°C until it is fully dried (maintain constant weight).
9. To begin the carbonization process, 200g of coconut coir is dried in oven at 700 °C in Fixed Bed Activation Unit (FBAU) with the presence of N_2 for 30minutes. The carbonization is vital in order to activate the coconut coir for arsenate removal.
10. The carbonized coconut coir it is washed with 200mL of 10% HCl followed by distilled water. CC is dried in oven overnight and kept in stoppered bottles.

3.2.3.1 Process Flow

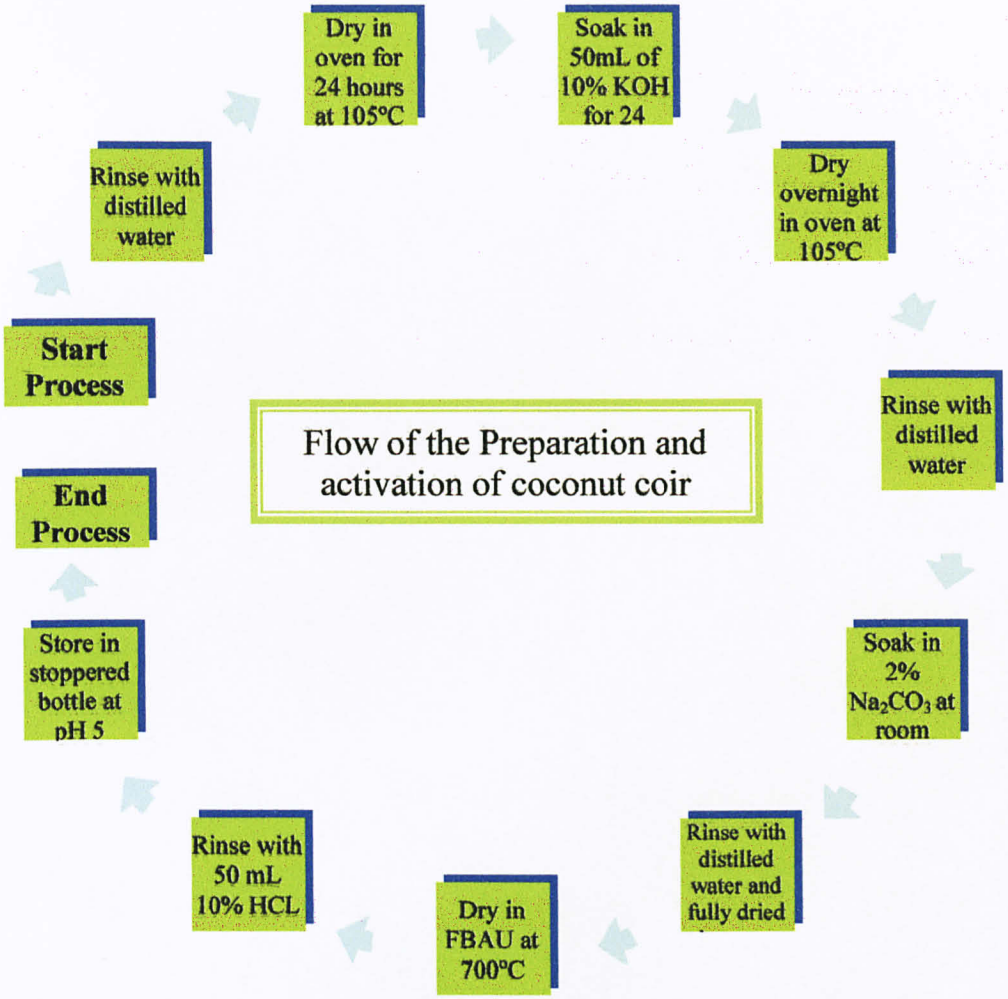


Figure 8 : Simplification of Activation process

Figure 8 shows the simplified activation process of the coconut coir. It was found that the dried coconut coir undergoes a reduction of weight after being dried in the oven for 15 minutes at 105°C. It is assumed that this reduction of weight was caused by evaporation of the moisture in the coir fibre. During this simple test, it was found that the moisture content in the coconut coir was around 52%. Figure 4 and 5 below illustrate the coconut coir that has been dried and treated.

(a) Before acid treatment b) After acid treatment

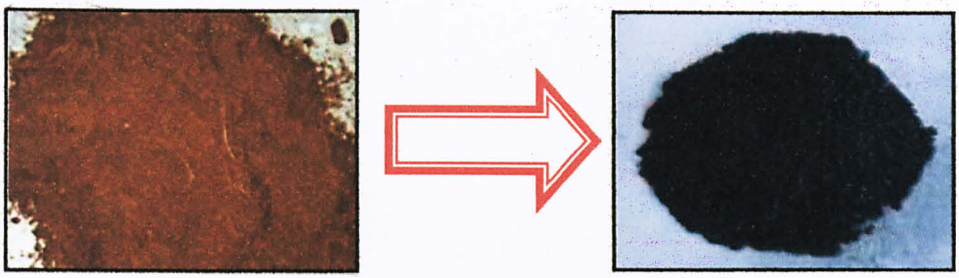


Figure 9 : Dried coconut coir before (a) and after (b) acid treatment

3.2.4 Preparation of 1000 ppm of Sodium Arsenate (Na_3AsO_4)

1. 1000mg Na_3AsO_4 is weighted by using electronic balance
2. 1000mg Na_3AsO_4 is placed in 1000mL volumetric flask and the flask is filled up with distilled water until the 1000mL level.
3. The solution is stirred with magnetic stirrer at 27°C ± 2 °C.

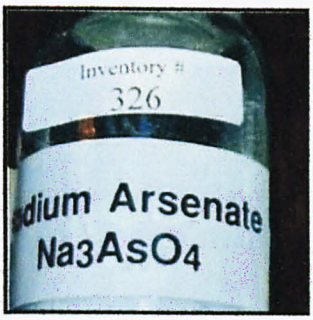


Figure 10 : Sodium arsenate in solid phase (salt)

Referring to Section 2.2 Potential Acute Health Effect, it is important to ensure to utilize all the essential Personal Protective Equipment (PPE).

3.2.5 Adsorption Kinetic Experiment

1. Five conical flasks of 500 mL with the concentration of 40 ppm of arsenate nitrate solutions are prepared by using the standard solution (1000 ppm).
2. The solutions is set at pH 5 are placed over mechanical shaker at 120 rpm at 27°C ± 2 °C. At time zero, 1.0 g of granular activated carbon is added to the respective conical flask.
3. Samples are taken at *(0.5, 1.0, 1.5, 2.0, 3.0, and 3.5 h)* and sampling is continued until the systems attain the equilibrium.
4. Solutions are filtered and analyzed using A.A.S.

3.2.6 Batch Adsorption Test

1. Five conical flasks of 100 mL arsenate nitrate solutions are prepared by using the standard solution (1000 ppm) at the following concentrations:
   Intial concentrations: 5 ppm, 20 ppm, 40 ppm and 100 ppm
2. The solutions is set at pH 5 are placed over mechanical shaker at 120 rpm at 27°C ± 2 °C. At time zero, 1.0 g of granular activated carbon is added to the respective conical flask.
3. 5 ml samples of each solution are withdrawn from the conical flask at known time intervals. Contact time will be used for batch tests.
4. Solutions are filtered and analyzed using A.A.S.

3.2.7 Effects of pH of Solution

1. 100 ml of 40 ppm arsenate nitrate solution is placed in a conical flask.
2. pH values of solution adjusted with 0.5M HCl and NaOH solutions :
   Varied pH: pH 2, pH 5, pH 7 and pH 8
3. The solution is placed on mechanical shaker at 120 rpm at 27°C ± 2 °C.
4. At time zero, 1.0 g of adsorbent is added into the conical flask.
5. 5 ml samples of solution are withdrawn from the conical flask at known time intervals.
6. Solutions are filtered and analyzed using A.A.S.

3.2.8 Effects of Amount of Adsorbent

1. 100 mL of 40 ppm Na_3AsO_4 solution is placed in a conical flask.
2. The mixture is set at pH 5 and placed over the mechanical shaker at 120 rpm at 27°C ± 2 °C.
3. At time zero, 0.2g of adsorbent is added into the conical flask.
4. 5 ml samples of solution are withdrawn from the conical flasks at known time intervals. The solutions are filtered and analysed.
5. The experiment is repeated with following amount of adsorbent :
   Amount of adsorbent: 0.2g, 0.8g, 1.0 g, 2.0g

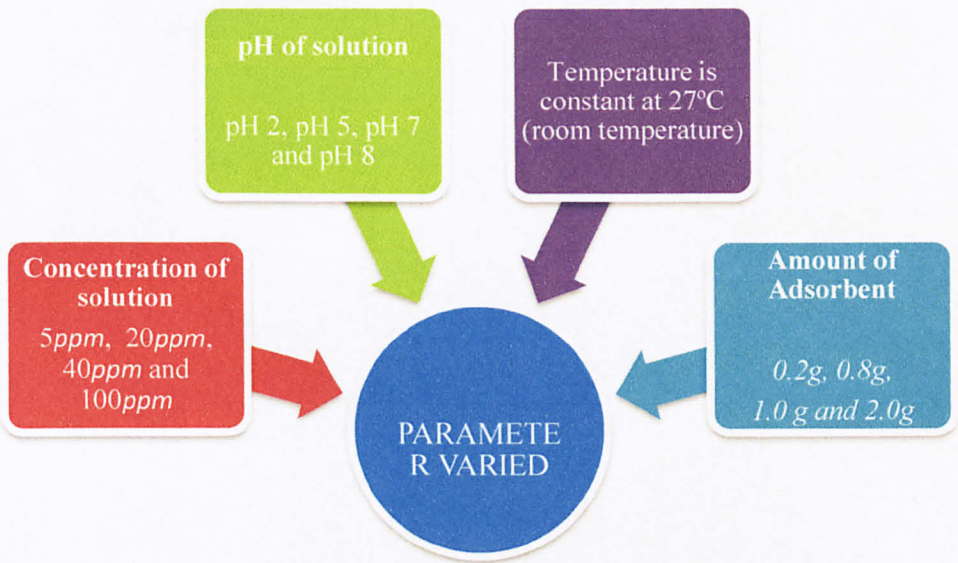


Figure 11 : Parameters of the experiments

3.3 Key Milestone

This project is divided into two semester where in Semester 1 is mostly on the research work meanwhile in Semester 2, the experimental work is been carried out. During Semester 1, the literature review and the methodology are explored based on previous research. In Semester 2, the laboratory work is conducted where all the parameters are varied accordingly. At the end of the semester, an experimental assessment is concluded.

3.4 Sources and Harwares Needed

3.4.1 Tools

a) Atomic Absorption Spectrophotometer (A.A.S)

The atomic absorption spectrometry uses absorption of light of intrinsic wavelengths by atoms. All atoms are classified into those having low energies and those having high energies [35]. The state having low energies is called the ground state and the state having high energies is called the excited state. The difference between energies in the ground state and in the excited state is fixed by the element and wavelength of light to be absorbed [36].

3.4.2 Apparatus

Oven, porcelain container/dish, furnace, Energy Dispersive X-ray Spectroscopy (EDX), Scanning Electron Microscope (SEM), X-ray Diffraction (XD), Fourier Transform Infrared Spectroscopy (FTIS), Thermogravimetric Analysis (TGA) device, mechanical shaker, bottle sample (100ml), pipette, cylinder measurement (1000ml), cylinder measurement (500mL), syringe (filtration), beaker (250ml), electronic balance, spatula, pH meter

3.1.1 Materials / Chemicals

Potassium hydroxide (KOH), sodium bicarbonate (Na_2CO_3), hydrochloric acid (HCl), distilled water, coconut coir, commercial activated carbon Merck, sodium hydroxide (NaOH), sodium arsenate (Na_3AsO_4)

CHAPTER 4
RESULTS AND DISCUSSIONS

The effect of contact time, initial concentration of adsorbate, pH of solution and amount of adsorbent on the arsenate adsorption were characterized in batch adsorption experiments. The choice of contact time for such experiments represents a result by exploring the true equilibrium condition and practical limitations.

4.1 Textural Characterization of AC

4.1.1 FTIR

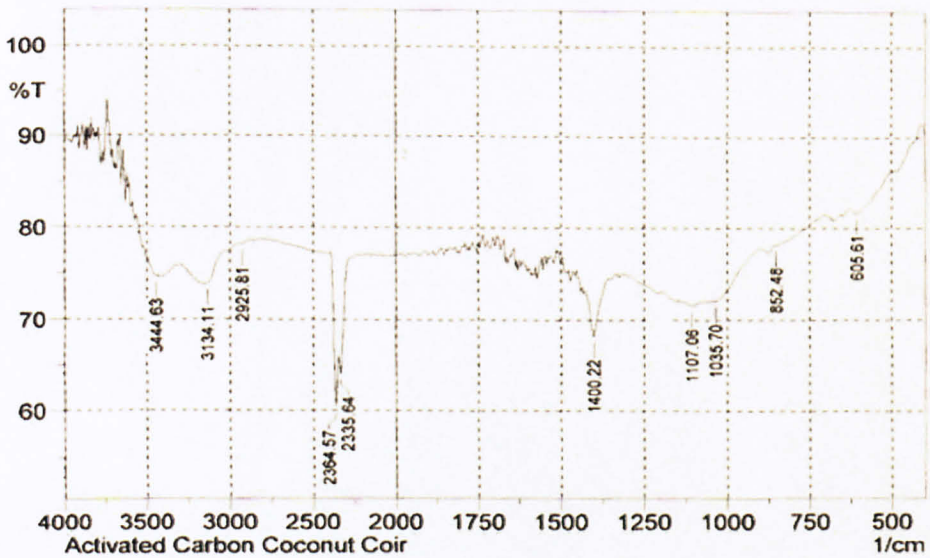


Figure 12 : FTIR spectrum on AC

A quantitative analysis of activated carbon was conducted by obtaining FTIR transmission spectra of carbon samples. The adsorption capacity of activated carbon depends upon porosity as well as the chemical reactivity of functional groups at the surface.

This reactivity creates an imbalance between forces at the surface as compared to those within the body, thus leading to molecular adsorption by the Van der Waals force. Knowledge on surface functional groups would give insight to the adsorption capability of the produced activated carbon.

FTIR spectra were collected for qualitative characterization of surface functional groups of porous carbons activated by both physical and chemical methods. Figure 12 shows the functional groups of activated carbon prepared from coconut coir.

The FTIR spectrum of coconut coir AC reveals a broad peak at 3444.63 cm^{-1} and 3134.11 cm^{-1} which can be attributed to the O-H group [25]. The peak observed at 2925.81 cm^{-1} corresponds to the C-H stretching group. The alkynes group of C≡C is found around 2364.57 cm^{-1} to 2335.64 cm^{-1}.

The vibration at 1400.22 cm^{-1} is assigned to the alkanes group of C-H. The peaks from 1035.70 cm^{-1} to 1035.70 cm^{-1} are assigned to the carbonyl stretching group, C-OH stretching in primary alcohol (1035.70 cm^{-1}) and secondary alcohol (1107.36 cm^{-1}) [40].

4.1.2 SEM

The long fibers of coir and the small pieces form coconut coir namely coconut coir dusts. For the characterization of CC by SEM these mixtures are examined. SEM gives the overblown 3D images with high resolution. The images are magnified with electrons instead of light waves for 100, 500 and 1000 times. The images show that there are quite a number of small pores that is important for adsorption.

Based on Figure 13, it shows that there are pores exist on the coconut coir fiber. These pores prove the capability of adsorption but these pores do not support maximum adsorption process.

Figure 13 : SEM images of I, II and III with the magnification of 50, 500 and 1000 respectively for coconut coir fiber before activation process

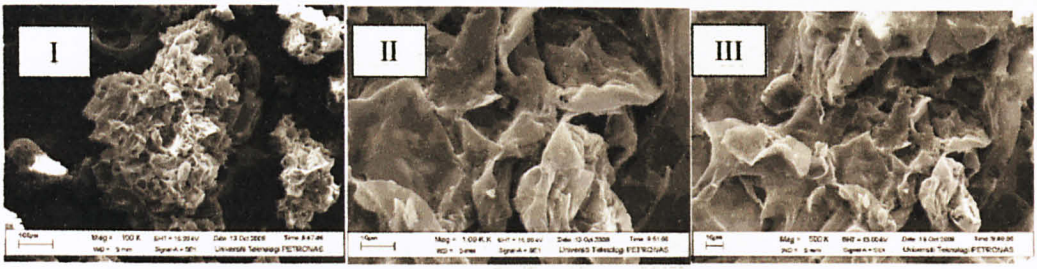


Figure 14 : SEM images of I, II and III with the magnification of 50, 500 and 1000 respectively for coconut coir dust before activation process

On the other hand, Figure 14 shows the structure of the dusts that are not uniform. There are also a lot of small pores or spaces inside the structure. These samples of coconut coir are before the acid activation and carbonization. Therefore it can be depicted that raw coconut coir is not suitable for the removal of arsenate via adsorption process.

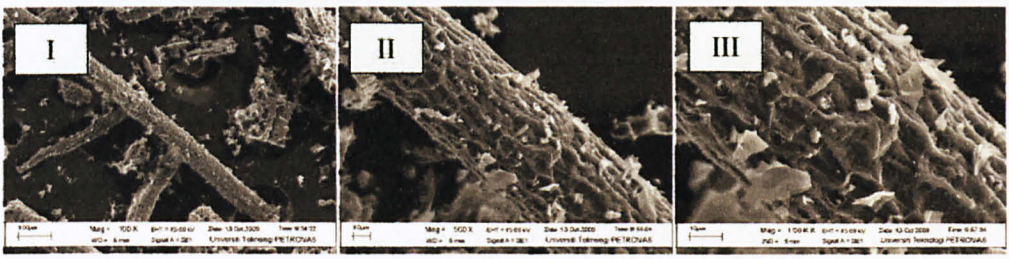


Figure 15 : SEM images of I, II and III with the magnification of 50, 500 and 1000 respectively for coconut coir fiber after activation process

After the acid treatment and carbonization process, the treated coconut coir samples are examined. Figure 15 and 16 shows the structure of the coconut coir fibers and dust respectively which have been converted into activated carbon.

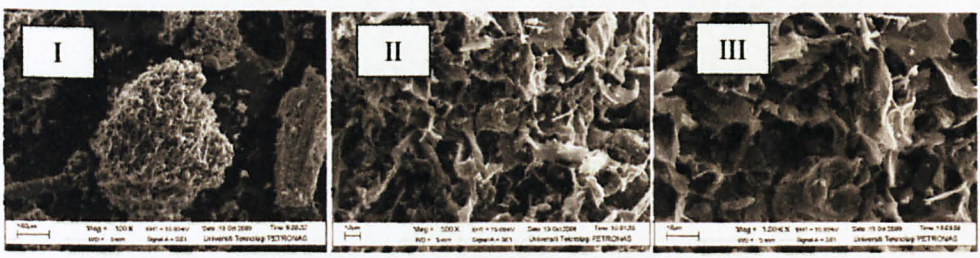


Figure 16 : SEM images of I, II and III with the magnification of 50, 500 and 1000 respectively for coconut coir dust after activation process

The number of pores of the coconut coir has been increased after the chemical and physical activation. These images show that the ability of the coir to adsorb is higher due to higher number of pores [41]. The structures have been more complex with more pores that forms chamber to capture foreign ions. Thus it will increase the capacity of adsorption

4.1.3 XRD

The crystal structure characteristics of the result samples are analyzed by x-ray diffractometer (XRD) as a radiation source. The measurement has been carried out in a scale 2θ and long duration scan 15s. The x-ray diffraction patterns of activated carbon sample are shown in figure above.

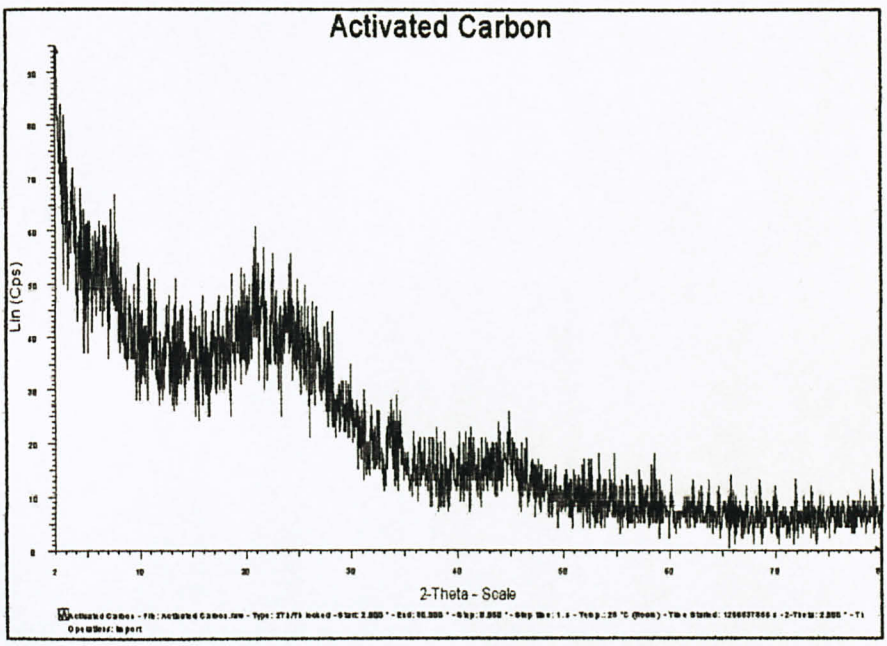


Figure 17 : X-ray diffractogram for activated carbon from coconut coir

Figure 17 shows a broad peak existed at $2\theta = 22^{o}$ with relative intensity of 58 cps for coconut coir correspond to the lattice plane cellulose. Reasonably, the peaks for the coconut coir increased with density which is commonly observed in nanosized carbon. The sharp lines superimposed on the broad carbon peaks are identified as silica, perhaps unavoidable impurity in activated carbon [41].

The cell walls of most of the natural fibers mainly consist of cellulose, hemicellulose, and lignin. Cellulose has both amorphous and crystalline regions, although hemicellulose and lignin are amorphous. The intensity of the peaks did change, suggesting differences in crystallinity and also their crystallite size depend on the heat treatment.

The XRD pattern of activated carbon fiber shows almost no diffraction lines and is confirmed to be the amorphous material [42]. The XRD pattern of result sample after synthesis shows the diffraction lines corresponding to cellulose in a face centered cubic structure.

Due to their thinness (or small "crystallite size") and the polycrystalline morphology of the coconut coir, their x-ray diffraction diagrams do not exhibit sufficiently detailed diffraction data to permit a full structure analysis to be completed, such as can be done with large single crystals [43]. The relative low temperature allows the formation of small amounts of stable structure which is evidenced by the peak located at around diffraction angle of 50^{o}.

In addition to the diffraction lines, a broad peak at low diffraction angles is observed corresponding to the presence of amorphous phase in the sample [44]. That could be attributing to the unreacted remaining carbon. When the coconut coir is treated with acids the majority of those peaks disappear due to leaching out the corresponding minerals during activation and washing with water.

4.1.4 TGA

A testing procedure in which changes in weight of a specimen are recorded as the specimen is heated in air or in a controlled atmosphere such as nitrogen. Thermogravimetric curves provide information regarding polymerization reactions, the efficiencies of stabilizers and activators, the thermal stability of final material and direct analysis.

The analyzer usually consists of a high precision balance with a pan generally platinum loaded with the sample. The pan is placed in a small electrically heated

oven with a thermocouple to accurately measure the temperature. The atmosphere is purged with inert gas to prevent oxidation and other undesired reactions. Computer is used to control the instrument.

Analysis is carried out by raising the temperature gradually and plotting weight against temperature. The temperature in set from 30°C till 350°C but the oven is greatly insulated that operator would not aware of any changes in temperature even if standing in front of the device.

After the data is obtained based on 5°C, 10°C and 15°C temperature variation, curve smoothing is done to find the exact point of inflection. Hi- res TGA is employed to obtain greater accuracy in areas where the derivative curve peaks. In this method, temperature increase slows down as the weight loss increases. This is done so that the exact temperature at which a peak occurs can be accurately identified.

Based on the Figure 18, 19 and 20, these are the result of coconut coir analysis at temperature variation of 5°C, 10°C and 15°C respectively. It is assumed that weight decreases with the increase of temperature due to the removal of structural water.

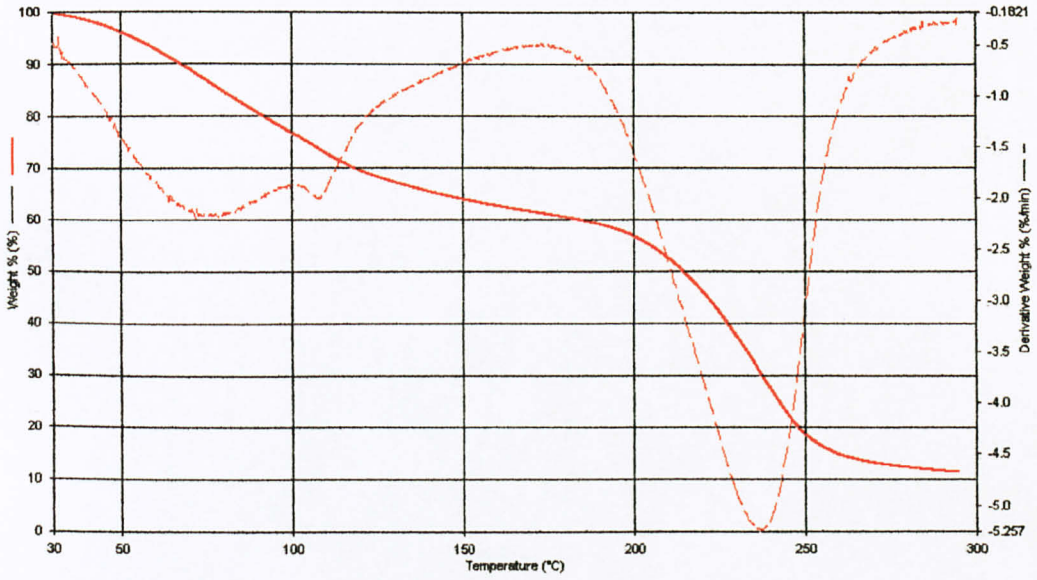


Figure 18 : TGA result at temperature variation of 5 °C

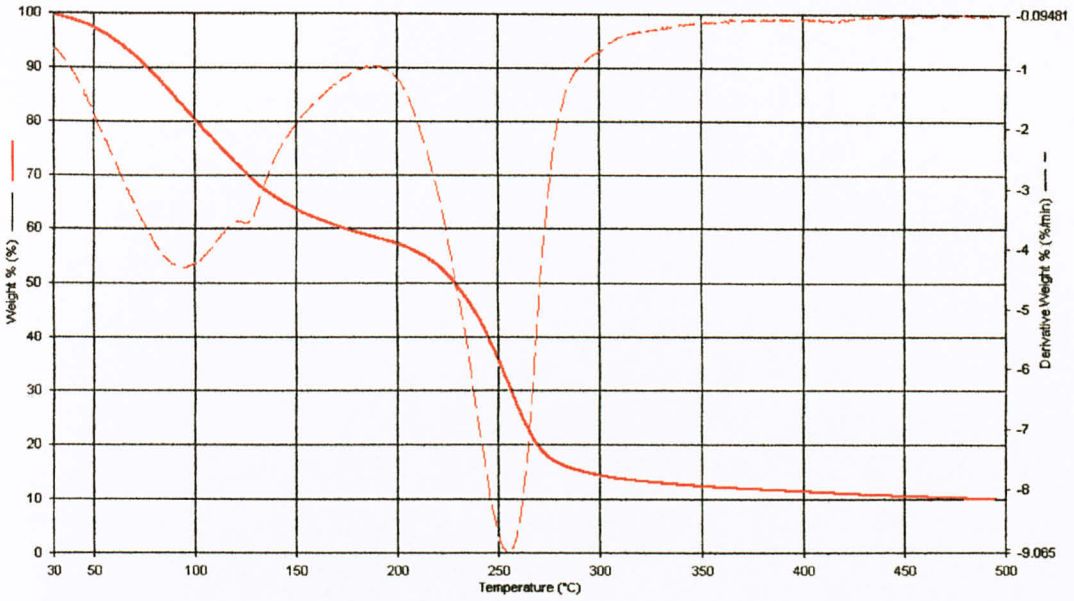


Figure 19 : TGA result at temperature variation of 10 °C

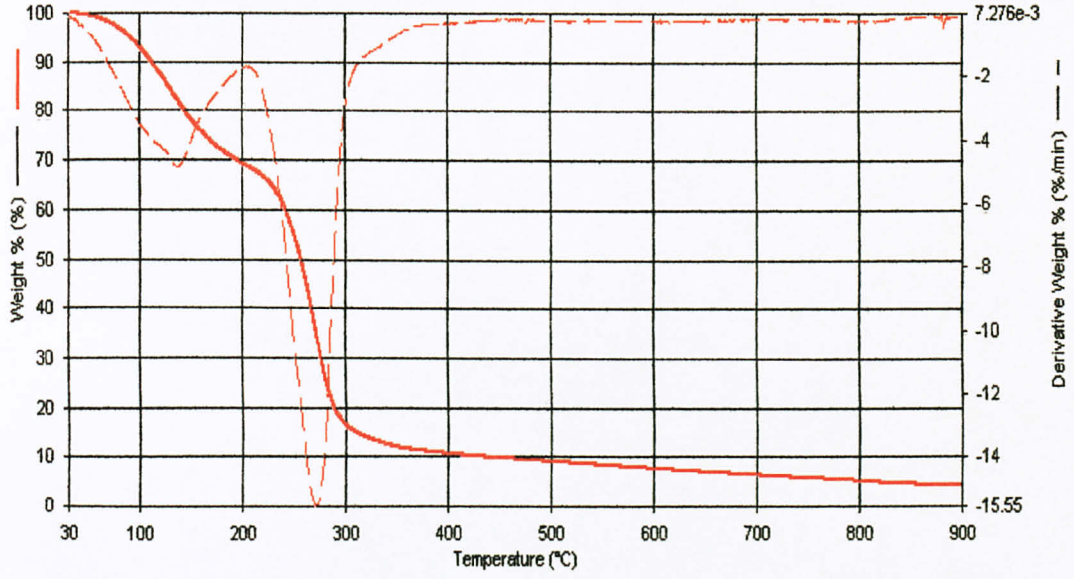


Figure 20 : TGA result at temperature variation of 15 °C

4.2 Adsorption Data Evaluation

The adsorption process towards removal of arsenate via adsorption process is evaluated based on contact time, effects of initial solution concentration, pH of solution and amount of adsorbent and the comparison of Merck activated carbon (AC) and activated carbon prepared from coconut coir (CC),.

The experiment is conducted using Na_3AsO_4 salt solution instead of pure arsenate solution due to safety purpose. As arsenic forms arsenate and arsenite in aqueous solution, adsorption of arsenate is focused compared to arsenite because arsenite in salt solution has been oxidized [11].

According to P. Roonasi (2009), Equation 1 (refer to pg.6), expresses the arsenate uptake capacity of adsorbent [42]. Apart from that, the arsenate uptake is best interpreted using Equation 2 to acknowledge the amount of lead uptake at time, *t* by calculating the difference between the initial and final concentration over the initial concentration, and multiplying it by a hundred [31].

The equation below calculates the optimum parameter (e.g. concentration) in each part of the experiment. Percentage of lead uptake at each instantaneous time interval, *t* conducted can be obtained from this. The data assessment in this project is by obtaining the values of percentages of arsenate uptake for effect of concentration and pH using Equation 3.

$$(\%)\ \text{Percentage of Uptake} = \frac{(C_0 - C_1)}{C_1} \times 100 \qquad \text{..... Equation 2}$$

Where

C_o is the initial concentration

C_1 is the final concentration

The adsorption of arsenate onto adsorbent can be explained with Van der Waals (VDW) forces which act as the decisive component of adsorption forces [32]. The VDW forces are the major contributor to the physical adsorption. Physical adsorption occurs when the arsenate ions are bounded onto the surface of the adsorbent by the weak VDW forces. This VDW forces act similarly to molecular cohesion [33].

4.3 Contact Time and Adsorption Kinetics

Preliminary kinetic experiments are conducted to access the time taken for the equilibrium to be attained. The concentration of arsenate solution is set at 40 ppm. Meanwhile, the temperature and pH of solution is held constant 27°C ± 2 °C and pH 5 respectively.

The mixture of solution and adsorbent is continuously stirred at 120 rpm. For evaluation in A.A.S., a sample of 5mL of the mixture is extracted at the time interval of 0.5, 1.0, 1.5, 2.0, 3.0, 3.5 h. Adsorption kinetics describes the solute uptake rate which controls the contact time (refer to section 3.2.5: Adsorption Kinetic Experiment).

The final arsenate ion concentrations obtained for batch experiments are converted to percentage of arsenate ion removed (% arsenate uptake) and amount of arsenate per unit weight of adsorbent (q_e). The data acquired is corrected accordingly assuming that the results are comprised after filtration. The trend for adsorption of arsenate using AC and the CC are shown in Figure 18.

Based on the Figure 18, adsorption onto the adsorbent was relatively rapid during the first 0.5h of exposure and slower thereafter. The significant removal of arsenate in at 0.5h for CC (62%) and AC (50%) leads on to no appreciable change in terms of removal after 1h. The final percentage of arsenate uptake for CC is 79% and AC 62%. The noticeable difference of removal after 0.5h is 17% for CC and 12% for AC.

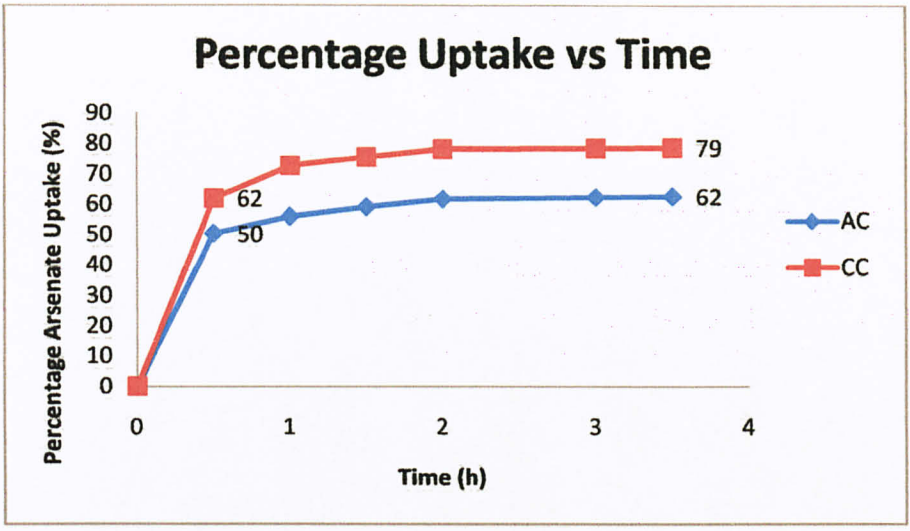


Figure 18 : Equilibrium time for the adsorption of arsenate on CC and AC

The equilibrium started to occur at 0.5h where adsorption process has begun. It indicates that initially, the active surface sites of the adsorbent are rapidly occupied with the arsenate ions. This is because in the beginning, active surface sites are vacant and readily available to occupy arsenate ions.

Arsenate ions interact easily with the active sites where more arsenate ions are adsorbed. In other words, more arsenate ions are removed from the aqueous solution [19]. The scenario of the rapid interaction between arsenate ions and adsorbent active sites increases the percentage of arsenate uptake onto the adsorbent. Therefore, the trend approaching 0.5h has higher slope indicating of rapid adsorption.

Towards 1h, the percentage of arsenate uptake starts to reduce and become constant till it reaches 4h, indicating that the process reaching the end of adsorption process. According to E. Emirbas (2004), this is due to the surface of adsorbent was saturated with the arsenate ions [16].

Since the amount of adsorbent used is constant at 1.0 g, the active surface sites are limited. Therefore, the adsorption of arsenate onto the adsorbent is also limited. The more arsenate ions accumulated onto adsorbents, the lower the numbers of vacant active sites, hereby reduces the percentage of arsenate uptake towards 1h.

Furthermore, the driving force for adsorption is the ratio between the bulk solution and the solid liquid interface. Initially, the bulk arsenate ions concentration is higher, meaning it enables higher adsorption rate. However, after the initial 0.5h period, the concentration of arsenate ions exist in the solution becomes lesser, giving a smaller ratio which results a flatter trend on Figure 18.

Lesser arsenate ions are being adsorbed onto the adsorbent giving a low percentage of arsenate uptakes. This is probably due to slower diffusion of solute onto the interior of the adsorbent as the surface of adsorbent is rich with arsenate ions [34].

Based on Figure 18, after the 1h, the trend of the adsorption curve is getting flatter and almost a straight line indicating that there is barely any adsorption towards 4h. Thus, the equilibrium time for the adsorption of arsenate onto the adsorbent is observed in the time frame of four hours.

In all subsequent experiments, the equilibrium time is maintained at 4h. The period of four hours (4h) is assumed to be sufficient for the removal of arsenate. The comparison between the two adsorbents will be discussed further in section 4.8.

4.4 Effects of Initial Concentration

The effects of concentration of the arsenate ions are observed by varying the arsenate concentrations in the range of 5 ppm, 10 ppm, 20 ppm, 40 ppm and 100 ppm. The temperature and pH of solution is held constant 27°C ± 2 °C and pH 5 respectively. The mixture of solution and adsorbent is continuously stirred at 120 rpm. For evaluation in A.A.S., a sample of 5mL of the mixture is extracted at the time interval of 0.5, 1.0, 1.5, 2.0, 3.0, 3.5 h.

Based on Figure 19, at 5 ppm, 20 ppm, 40 ppm and 100 ppm, the percentage of arsenate uptake are 99%, 95%, 92% and 69% respectively. It is obvious that the percentage of arsenate uptake is higher when the concentration is increased from 5 ppm to 40 ppm. The average value of percentage uptake in the range of 5 ppm to 40 ppm is approximately 95%.

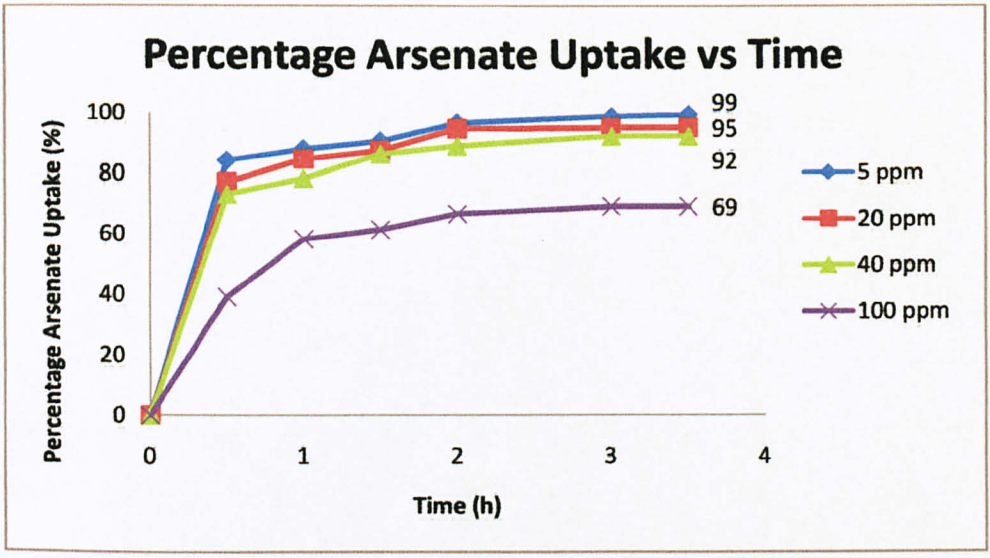


Figure 19 : Effects of initial solution concentration on adsorption of arsenate onto CC; 1g of CC

As mentioned by Amaransinghe (2007), at low arsenate concentrations, the ratio of surface active sites to the total arsenate ions in the solution is high [19]. Hence, the arsenate ions may interact rapidly with the adsorbent and be removed from the solution. Consequently, it increases the percentage of arsenate uptake onto CC over time.

On contrary, by increasing the concentration to a very high value (100 ppm), the percentage of arsenate uptake is reduced dramatically. The percentage of arsenate uptake reduces from 92% to 69%, giving a difference of -23%. This is probably due to the ratio of surface active sites to the total arsenate ions exists in solution.

At higher arsenate concentration, the ratio of surface active sites are relatively lower to the total arsenate ion available in the medium of solution, hereby, the surface active sites are immediately occupied, resulting a saturated surface area which delays the arsenate adsorption onto the CC [19].

Apart from that, the amount of adsorbent used (1.0 g) may not be sufficient to support a very high amount of arsenate ions. Therefore, the amount of arsenate ions adsorbed onto the adsorbent is limited. Referring to Figure 19, at 5 ppm, the percentage of arsenate uptake is 99% where else at 100 ppm is 69%. Though, compared to the amount of arsenate adsorbed at 5 ppm, the value is only 2.48 mg/g where else at 100 ppm, the values is 39.57 mg/g.

It can be deduced that at higher concentration, the excessive arsenate ions are not being adsorbed onto the CC. This is due to the initially vacant active surface sites are saturated with adsorbed arsenate ions. According to Adbel-Halim S.H., the higher the initial concentration, the higher the arsenate ions in solution, results a lower percentage uptake of arsenate via adsorption [37]. It is proven by the percentage of arsenate difference of 30% between 5 ppm and 100 ppm in Figure 19.

However, amount of arsenate adsorbed per unit weight of adsorbent, q_e (refer to Equation 1) is higher at high concentration as shown in Figure 20. At 100 ppm, the q_e is 39.57 mg/L, which is 93% higher compared to q_e at 5 ppm. Even though the percentage of arsenate uptake at 100 ppm is lower, the amount of arsenate adsorbed is the highest.

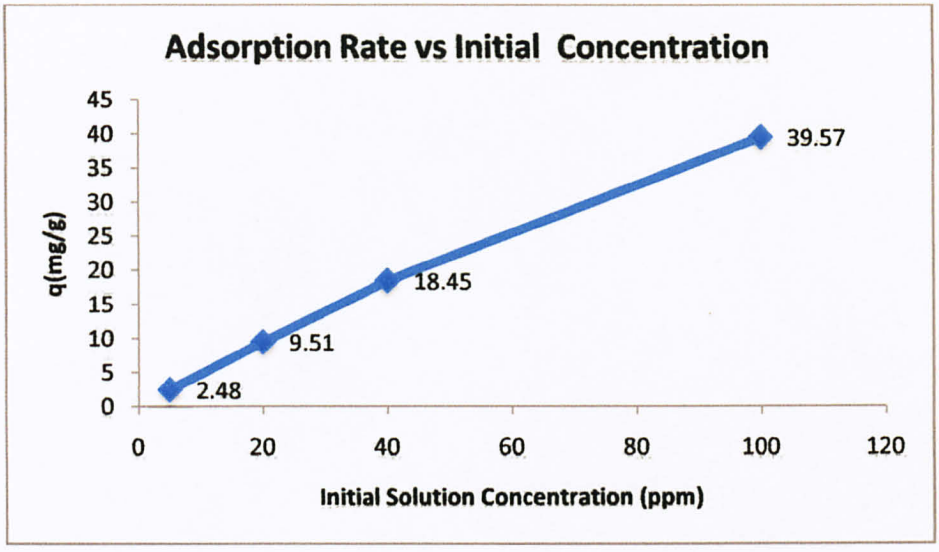


Figure 20 : Absolute amount of arsenate adsorbed onto CC; 1g CC

Referring to Rajeev Goel (2001), the higher amount of arsenate ions in the solution, more arsenate ions are capable to be adsorbed onto the CC. Therefore, q_e is significantly higher. It is proven that the initial concentration of the solutions affects the adsorption capacity of the arsenate ions onto CC. (All the data and calculation involved are presented in Excel spreadsheet attached in the Appendix)

4.5 Effects of pH of Solution

The adsorption of arsenate is studied over the pH range 2, 5, 7 and 8. The concentration of arsenate solution is set at 40 ppm. The temperature of solution is held constant 27°C ± 2 °C. The mixture of solution and adsorbent is continuously stirred at 120 rpm. For evaluation in A.A.S., a sample of 5mL of the mixture is extracted at the time interval of 0.5, 1.0, 1.5, 2.0, 3.0, 3.5 h. The range is below pH 8.0 to avoid chemical precipitation. The objective is to study the adsorption process, thus the chemical precipitation due to high pH value is neglected.

Figure 21 shows the adsorption percentages for CC under varying pH conditions with constant level of arsenate and amount of adsorbent.

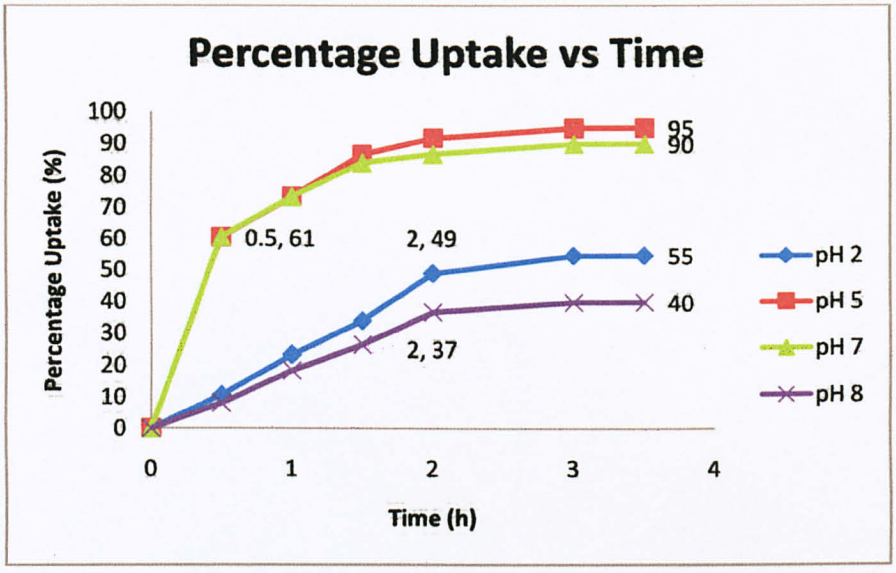


Figure 21 : Effects of pH on the adsorption of arsenate onto CC; 1g of CC

Arsenate adsorption is somewhat sensitive to pH in the range investigate. Referring to Figure 21, at pH 2, 5, 7 and 8, the percentage of adsorption are 55%, 95%, 90% and 40 % respectively. At pH 2, the percentage of adsorption is very low and rapidly increases between pH 5 and 7. In the pH range of 5 and 6, the percentage uptake of arsenate can be optimum (95%). By increasing pH from 7 to 8, it decreases the adsorption percentage by approximately 50%.

Based on the adsorption trend in Figure 21, the significant trend for pH 5 to pH 7 is noted where the trends reach equilibrium at quickly at 0.5h compared pH 2.0 and 8.0 at 2h. This phenomenon can be explained by the surface charge of the adsorbent and the H^+ ions present in the solution [21]. The result is partly influenced by the effect of carbonization and chemical activation stages conducted prior to experiments conducted.

The carbonization process conducted to the CC in this experiment was done by burning (carbonizing) the coconut coir at 700°C in the FBAU (refer to Section 3.2.3: Simplification Activation Process). As mentioned by Kobya M. and E. Emirbas (2005), the carbonization process provides an opportunity residual elementary carbon to be stacked randomly in cross-linked manner, creating free interstices in between atoms of carbon [38].

As quoted by Sekar, Sakth and Rengaraj (2004), during carbonization, the material is converted to active carbon [39]. Moving on to the chemical activation process, the carbonized coconut coir is chemically activated in 10% of HCl which contributes to larger pores with extended and extremely high surface area.

At low pH, CC is positively charged and at high pH, it is negatively charged. At pH 2.0, the adsorption percentage (55%) is very poor. Referring to Kobya M. (2005), it is due to protonation (positively charged) [38]. At pH lower than 5, the carbon surface of the adsorbent experience excessive protonation which results a decrease in adsorption of arsenate.

Therefore, it results a lower percentage of adsorption. A similar influence of pH has been reported by Rajeev Goel and Viviek Kumar (2001), [22]. The surface of adsorbent for CC changes its polarization according to the external pH [34]. The pH of adsorbent somehow controls the attraction of metal ions onto its surface active sites.

In contrast, at very high pH values, the metal complex forms and results precipitation and therefore, the separation may not be due to adsorption [35]. Hence, the adsorption of arsenate onto CC is at optimum in pH range 5.0 - 7.0. It explains the appreciable low value of percentage of arsenate uptake on CC at pH 8 (40)

Figure 22 shows the percentage of arsenate ion adsorbed on to the CC as a function of pH. The maximum removal or arsenate ions from aqueous solution is at pH 5 (92%) and pH 7 (90%). To the contrary, at pH 2, the percentage of arsenate uptake onto CC is only 55%, and dramatically low at pH 8 (40%).

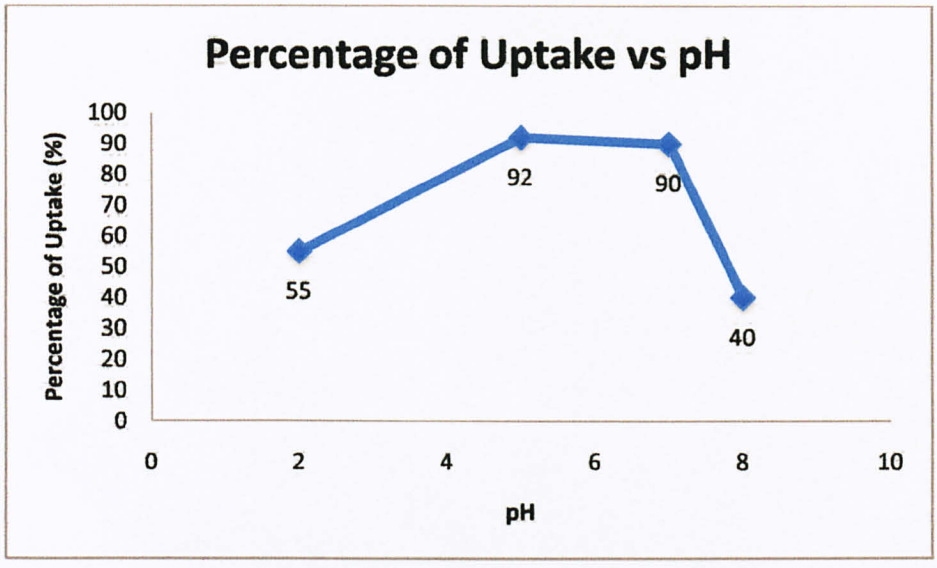


Figure 22 : Effect of pH on the adsorption of arsenate by CC; 1g of CC

It is clearly illustrated by Figure 22 that adsorption of arsenate ions onto CC is affected by the pH solutions. At a very acidic (pH 2) and a very alkaline (pH 8) environment, the adsorption of arsenate is not optimized. The maximum adsorption occurs in the range of pH 5 and pH 7.

4.6 Effects of Amount of Adsorbent

The effect of amount of adsorbent on removal of arsenate is studied by varying the amount of adsorbents at fixed arsenate concentration. The amount of adsorbents used in this study is varied from 0.2 g, 0.8 g, 1.0g and 2.0 g. The concentration of arsenate solution is set at 40 ppm. The temperature and pH of solution is held constant 27°C ± 2 °C and pH 5 respectively. The mixture of solution and adsorbent is continuously stirred at 120 rpm. For evaluation in A.A.S., a sample of 5mL of the mixture is extracted at the time interval of 0.5, 1.0, 1.5, 2.0, 3.0, 3.5 h.

Figure 23 shows the percentage of arsenate uptake onto CC at 4 varied amount of adsorbent used for constant value of initial concentration and pH of solution. Even though different amount of adsorbent is used, the equilibrium time for each adsorption process is maintained at 4h.

The percentage of arsenate uptake is increased from 15% to 95% when the adsorbent amount is increased from 0.2 g to 1.0 g. However, the percentage of arsenate uptake onto CC ironically reduces to 58% when the amount of adsorbent is increased to 2.0 g, providing a difference of approximately 40%.

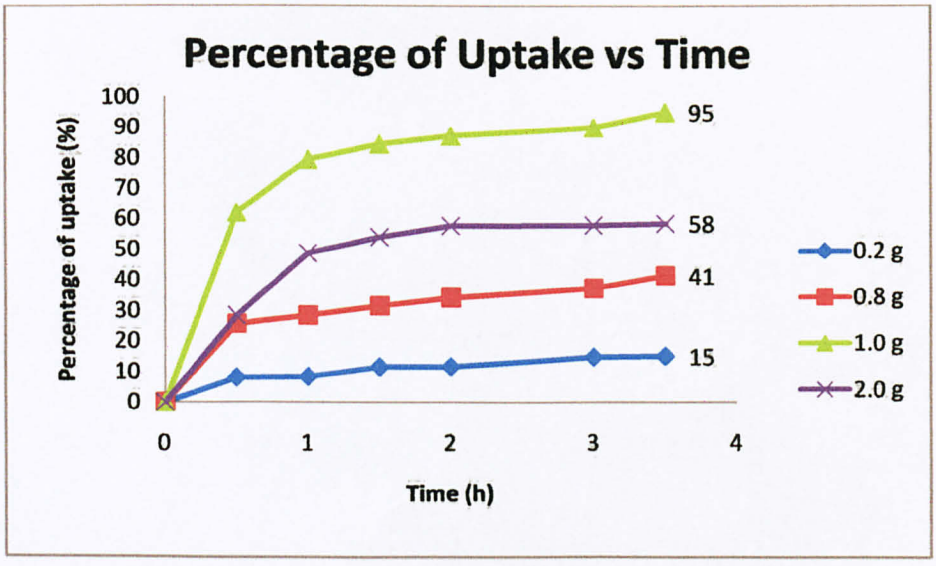


Figure 23 : Effects of adsorbent amount on the adsorption of arsenate on CC; 1g CC

Based on the adsorption trend in Figure 23, the highest percentage of arsenate uptake onto CC is at 1.0 g adsorbent with the value of 95%., indicating that the amount of 1.0 g adsorbent is sufficient for maximum removal of arsenate.

Referring to Longfei Lu, (2009) [45], the amount of adsorbent plays a big role in adsorption process. The amount of adsorbent determines the number of active surface area [40]. As the amount of adsorbent increases, the number of active surface area is multiplied. Therefore, more arsenate ions are possibly adsorbed onto the vacant surface area. Hereby, the percentage of arsenate uptake is increased, as the amount of adsorbent is increased.

The lowest percentage of arsenate uptake is at 0.2 g. This is due to the minimum number of active surface area given the small amount of adsorbent. The ratio of arsenate ions to the available active surface area is very high [41]. Hereby, the competitions between arsenate ions are higher, affecting the percentage of arsenate uptake.

As the amount of adsorbent increases, the numbers of active surface sites are augmented, instantaneously increases the amount of arsenate adsorbed onto CC. However, a very high amount of adsorbent leads to coagulation where the adsorbent forms clot where it reduces the surface area in total [44]. Hence, the percentage of arsenate uptake is reduced distinctly to 58%.

Figure 24, the amount of arsenate ions adsorbed per unit weight of adsorbent (q) decreases with the adsorbent dose. The highest amount of arsenate adsorbed onto CC is 18.98 mg/g (at 1.0 g) where else the lowest is 2.99 mg/g (at 0.2 g). At the highest amount of adsorbent (2.0 g), the amount of arsenate adsorbed is only 11.68 mg/g. It is assumed that coagulation of activated carbon defies the adsorption process. Hence, 1.0 g amount is selected as optimized amount to carry out successive batch experiment.

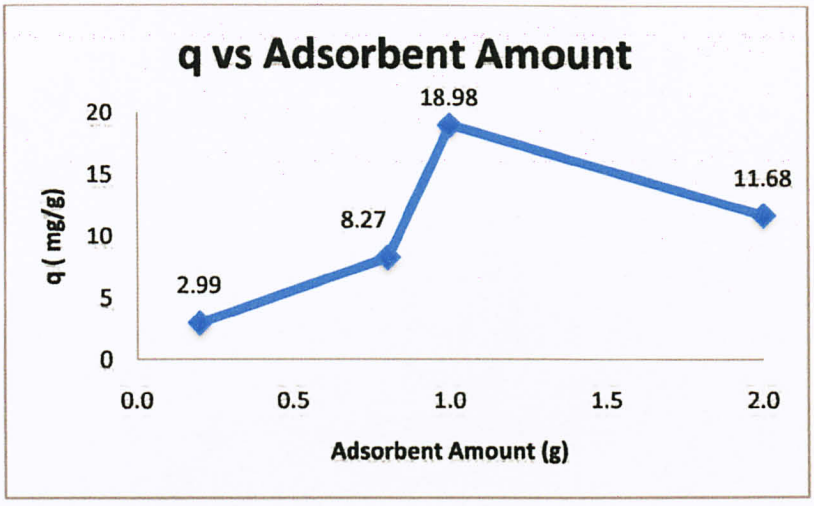


Figure 24:The absolute amount of arsenate adsorbed by the CC

Figure 24 demonstrates that adsorption of arsenate ions onto CC is affected by the amount of adsorbent used. The maximum adsorption occurs when 1.0 g of adsorbent is used. The adsorption is not favoured when the amount of adsorbent is very low (0.2 g) or very high (2.0 g). Thus, it can be concluded that the optimum amount of adsorbent need for removal of arsenate ions from aqueous solution is 1.0 g.

4.7 Comparison of CC and AC

Activated carbon is commonly used adsorbent in adsorption practice. Experiments were conducted to compare the adsorption characteristic of CC and AC. The concentration of arsenate solution is set according to Table 2. The temperature and pH of solution is held constant 27°C ± 2 °C and pH 8 respectively. The mixture of solution and adsorbent is continuously stirred at 120 rpm. For evaluation in A.A.S., a sample of 5mL of the mixture is extracted at the time interval of 0.5, 1.0, 1.5, 2.0, 3.0, 3.5 h. The result presented in Table 2 comprises only 4 experiments to compare the adsorption of arsenate onto AC and CC.

Table 2 : Comparison of arsenate adsorption onto AC and CC

Adsorbent	AC		CC	
Experiment Data	D1	D2	D3	D4
Adsorbent dose (g)	0.2	1.0	0.2	1.0
Initial Solution Concentration (ppm)	40	100	40	100
Arsenate Percentage Uptake (%)	62	80	79	69
Amount Adsorbed (mg/L)	12.5	40.08	15.7	39.7

By comparing D1 and D3 with the initial concentration value of 40 ppm and 0.2g of adsorbent per 100 mL solution of arsenate, it shows that D3 (79%) has higher percentage of arsenate uptake compared to D1 (62%). The adsorption of arsenate onto CC is higher then AC for the most of the experiments compared to AC except at concentration 100mg/L.

On contrary, at the initial concentration value of 100 ppm, the percentage of arsenate uptake for AC (80%) is higher than CC (69%). The amount of arsenate for D2 (40.08 mg/L) is slightly more than D4 (39.7). This scenario is probably because at higher concentration, due to excessive arsenate ions in the solution, most of the active surface sites of CC have been occupied with arsenate ions.

Thus, the ratio of saturated active surface sites to the vacant active surface sites is lower. The lower ratio indicates a lower percentage of arsenate uptakes which immediately results a lower value of arsenate removed from the aqueous solution via adsorption process.

Based on Figure 18, the trends are quite similar between AC and CC. Although the trends are quite comparable, arsenate adsorbs to a significantly greater extend on CC than on AC. Percentage uptake of arsenate onto CC is 79% where else percentage of arsenate onto AC is 62%. Percentage of arsenate uptake on CC is 17% more compared to the percentage of arsenate uptake onto AC.

According to N.R Bishnoi and M. Bajaj (2008), higher percentage of adsorption is probably due to higher surface area of CC compared to AC. Due to the lower surface area of AC, there may be no apparent diffusion [39] of arsenate to the interior of AC which influences the adsorption of arsenate onto AC. Thus, the percentage of arsenate uptake onto AC is relatively lower than onto CC [34-36].

Despite the corresponding adsorption capacity between AC and CC, CC can be obtained cheaply in large scale compared to the AC. Consequently, removal of arsenic from aqueous solution via adsorption process can be done vastly given the lower cost of adsorbent.

Figure 25 shows the comparison of adsorption capacities of arsenate on AC and CC. Adsorption capacities on CC is higher compared to AC except for the concentration 100 mg/L where the AC has higher (40.08 mg/g) adsorption rate compared to CC (39.57 mg/g). As mentioned earlier, a higher percentage of arsenate uptake for AC at 100 mg/L is probably also contributed by to the higher surface area of AC.

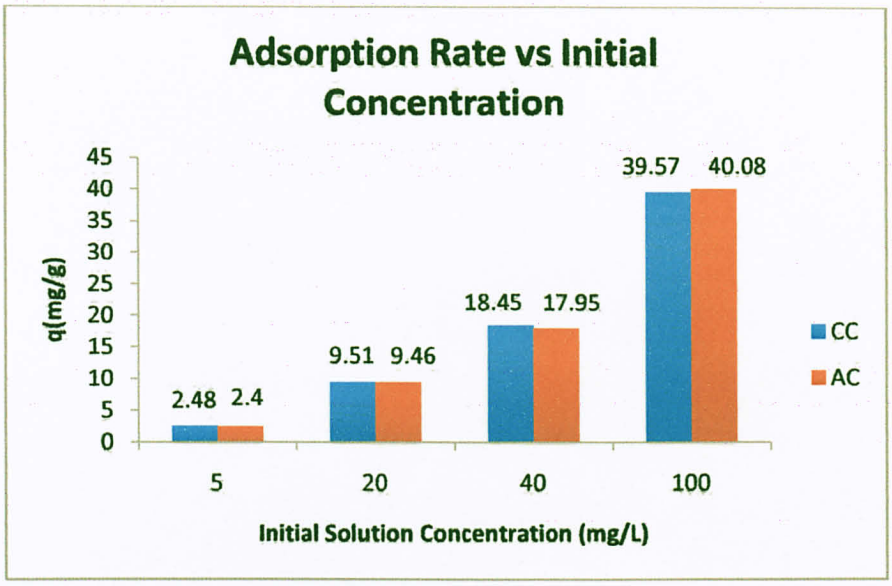


Figure 25 : Comparison of adsorption capacities of arsenate onto CC and AC; 1g of CC

Based on the near identical result in Figure 25, it can be say that CC may be a perfect alternative to replace the expensive commercialized activated carbon. Thus, it can be concluded that CC has potential to be explored as activated carbon since it has a similar adsorption capacity compared to AC. Besides, CC is far more economically viable compared to AC.

4.8 Summary of Discussion

The results obtained clearly demonstrated the potential use of CC for the removal of arsenate from aqueous solution. These are the following end result;

i) The kinetic studies indicated that equilibrium in the adsorption of the arsenate onto the CC was reached at 0.5h of contact between the CC in the arsenate solution (refer to Figure 18). After the 0.5h reaching 4h, there is no appreciable change. It is assumed that equilibrium time is attained at 4h for all the experiments, where the removal of arsenate is sufficient in 4hours.

ii) The optimum concentration of arsenate solution that favours the adsorption process is at 40 ppm. The percentage of arsenate uptake is increased with an increase in concentration of the arsenate solution (refer to Figure 19). However, at a very high concentration of arsenate solution, the percentage of arsenate uptake is ironically reduced due to the excessive amount of arsenate ions compared to the numbers of vacant active surface sites.

iii) The absolute amount of arsenate adsorbed onto CC is increased with the increase of concentration (refer to Figure 20). This is because as higher the concentration of arsenate solution, the amount of arsenate ions exists in the solution escalates.

iv) The optimum pH corresponding to the maximum adsorption was found to lie at pH 5 (refer to Figure 21). The selection of pH range is in between pH 2 and pH 8. At pH below 2, adsorption is impossible due to the protonation meanwhile at pH above 8, chemical precipitation forms metal complex which is no longer considered removal via adsorption.

v) The percentage of arsenate uptake onto CC is increased along with an increase of the amount of adsorbent (refer to Figure 23 and 24). The ideal amount of adsorbent that maximize the adsorption is 1.0 g. When the amount of adsorbent is increased to 2.0 g, the adsorption of arsenate is dramatically affected due to the coagulation of CC that potentially reduces the surface area of CC.

vi) The percentage of arsenate uptake on CC and AC gives two near identical trend. However the percentage of arsenate uptake for CC is slightly higher than AC. (refer to Figure 25)

CHAPTER 5
CONCLUSION AND RECOMMENDATION

CC is a good adsorbent for removal of arsenate from aqueous solution. CC shows higher adsorption capacity and rate compared to AC (refer to Section 4.2). The adsorption rate was rapid over an initial period of time and then decreases gradually. The percentage of arsenate ion uptakes and the adsorption capacity depend on the initial solution concentration, pH of solution, and amount of adsorbent.

The initial concentration of solution, pH of solution and the amount of adsorbent used are the main operating conditions that affects the adsorption capacity and percentage of arsenate ions uptakes onto adsorbent in a 4h contact time. The equilibrium time for the adsorption of arsenate ions from the aqueous solution onto adsorbent is attained at 0.5h.

Based on the experimental assessment, the adsorption is optimized at the initial concentration of 40 ppm, at pH 5 and the amount of adsorbent used is 1.0g. By comparing AC and CC, CC has been proven to have a better or equally good adsorption capacity as AC. Adsorption is rapid for CC compared to AC.

In this project, CC has been treated chemically and thermally where it is applicable as an alternative for the commercialized AC. The potential of CC as an adsorbent for the removal of arsenic from aqueous solution has been discovered. Since CC is economically viable, it is recommended to focus more to improve its adsorption capacity.

For further studies, it is recommended to investigate the effect of temperature of solution towards adsorption of arsenate. It is important to find out the optimum temperature for the effective adsorption process in order to manipulate the adsorption process. Besides, it is essential to determine the adsorption capacity when arsenic forms arsenate and arsenite in water, presumably arsenite influences the adsorption of arsenate.

In short, considering the high capability of coconut coir as an adsorbent of arsenic via adsorption, it is imperative to encourage more research by manipulating the coconut coir waste disposal crisis in order to resolve the pollution of arsenic in aqueous solution.

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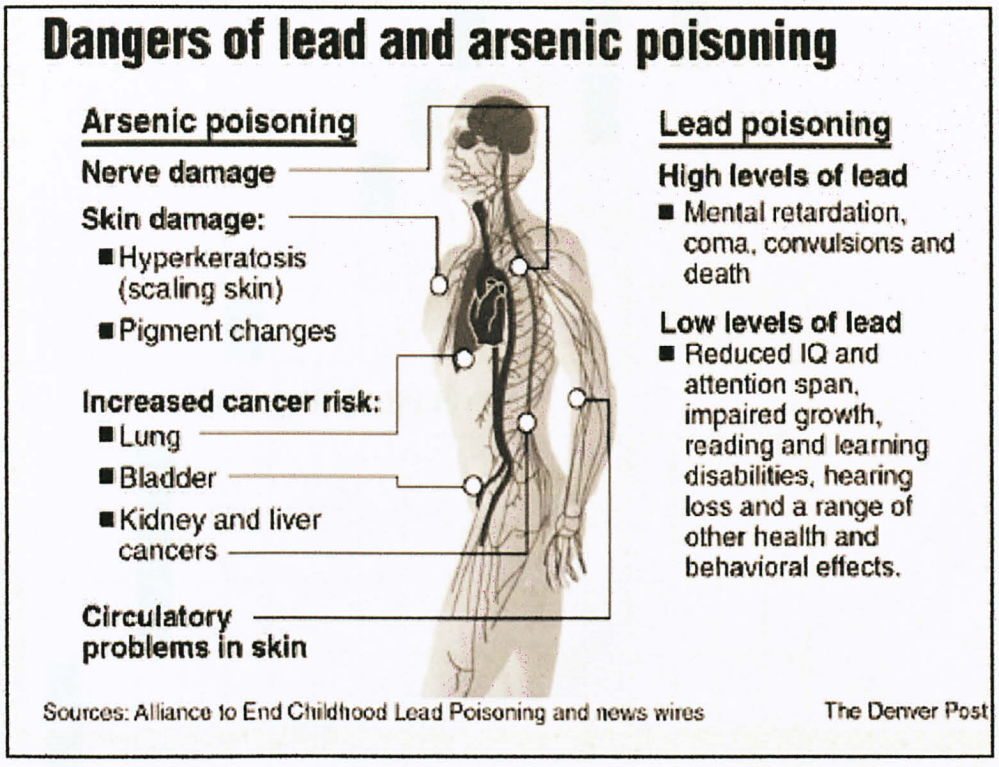
APPENDICES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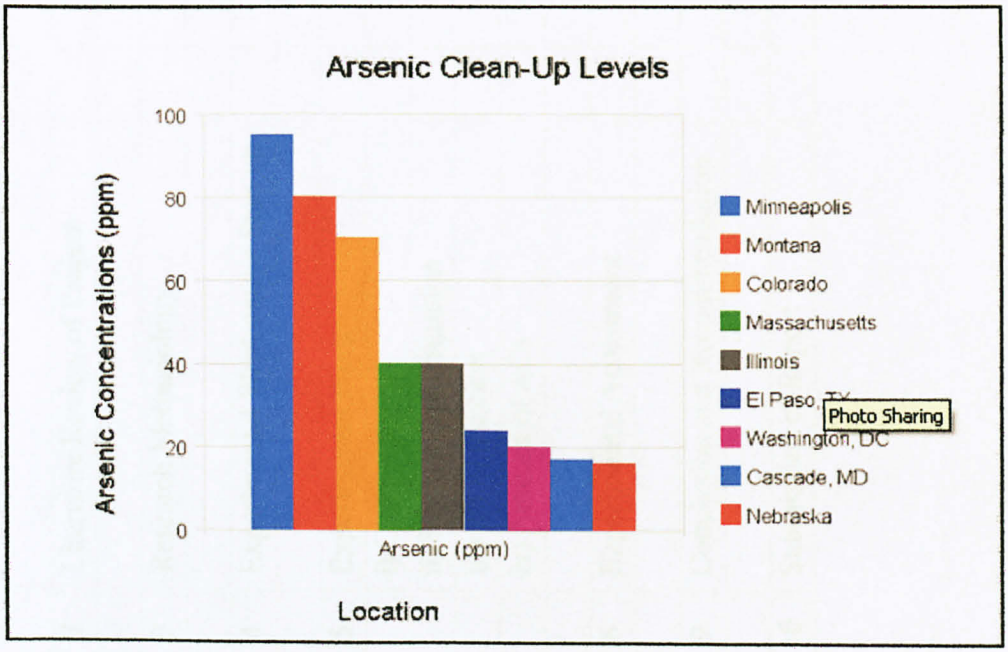
Arsenic poisoning
Nerve damage
Skin damage:
Hyperkeratosis (scaling skin)
Pigment changes
Increased cancer risk:
Lung
Bladder
Kidney and liver cancers
Circulatory problems in skin
Lead poisoning
High levels of lead
Mental retardation, coma, convulsions and death
Low levels of lead
Reduced IQ and attention span, impaired growth, reading and learning disabilities, hearing loss and a range of other health and behavioral effects.
Sources: Alliance to End Childhood Lead Poisoning and news wires
The Denver Post


Arsenic Clean-Up Levels
Arsenic Concentrations (ppm)
100
80
60
40
20
0
Arsenic (ppm)
Location
Minneapolis
Montana
Colorado
Massachusetts
Illinois
El Paso, TX
Washington, DC
Cascade, MD
Nebraska
Photo Sharing

PROJECT GANTT CHART

No	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Identification of Objective															
2	Literature Review of Project															
3	Research Methodology															
4	Experimental Workstation Start up															
5	Experimental Work															
	i) Contact time															
	ii) Solution concentration															
	iii) pH of solution															
	iv) Amount of ACs															
6	Experimental Assessment															
9	Conclusion and Recommendation															
10	Submission of Report															

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Health	4
Fire	0
Reactivity	1
Personal Protection	E

Material Safety Data Sheet
Sodium Arsenate Heptahydrate MSDS

Section 1: Chemical Product and Company Identification

Product Name: Sodium Arsenate Heptahydrate

Catalog Codes: SLS4546

CAS#: 10048-95-0

RTECS: CG3675000

TSCA: TSCA 8(b) inventory: Sodium arsenate

CI#: Not available.

Synonym: ; Arsenic acid, disodium salt, heptahydrate; sodium acid arsenate heptahydrate; Disodium arsenate, heptahydrate; Sodium arsenate, dibasic, 7-hydrate

Chemical Name: SODIUM ARSENATE HEPTAHYDRATE

Chemical Formula: Na2HAsO4 7H2O

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CHEMTREC (24HR Emergency Telephone), call: 1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Sodium Arsenate Heptahydrate	10048-95-0	100

Toxicological Data on Ingredients: Arsenic Acid, Disodium Salt (7778-43-0): No LD50/LC50 information found relating to normal routes of occupational exposure. Investigated as a mutagen.

Section 3: Hazards Identification

Potential Acute Health Effects:
Inhalation:
Arsenic may cause inflammation of the mucous membranes with cough and foamy sputum, restlessness, dyspnea, cyanosis, and rales. Symptoms like those from ingestion exposure may follow. May cause pulmonary edema.
Ingestion:
Arsenic is highly toxic! May cause burning in esophagus, vomiting, and bloody diarrhea. Symptoms of cold and clammy skin, low blood pressure, weakness, headache, cramps, convulsions, and coma may follow. May cause damage to liver and kidneys. A suspected fetal toxin. Death may occur from circulatory failure. Estimated lethal dose 120 milligrams.
Skin Contact:
May cause irritation, symptoms including redness, itching, and pain.
Eye Contact:
May cause irritation with itching, burning, watering of eyes; may cause conjunctiva damage.

Effect of Initial Arsenate Solution Concentration (CC)

t	time
Co	Initial Concentration
q	As Uptake
%	Percentage of As Uptake

Volume= 0.5 L
AC= 1g
Co = 40 mg/L

Co of 5ppm of AsNaO2			
t	Co	q	%
0	5.000	0.000	0
0.5	0.786	2.107	84
1	0.597	2.202	88
1.5	0.467	2.267	91
2	0.168	2.416	97
3	0.067	2.467	99
3.5	0.035	2.483	99

Co of 20 ppm of AsNaO2			
t	Co	q	%
0	20.000	0.000	0
0.5	4.579	7.711	77
1	3.045	8.477	85
1.5	2.458	8.771	88
2	1.046	9.477	95
3	0.989	9.505	95
3.5	0.975	9.512	95

Co of 40 ppmof AsNaO2			
t	Co	q	%
0	40.000	0.000	0
0.5	10.786	14.607	73
1	8.667	15.667	78
1.5	5.418	17.291	86
2	4.351	17.825	89
3	3.114	18.443	92
3.5	3.104	18.448	92

Co of 100 ppm of AsNaO2			
t	Co	q	%
0	100.000	0.000	0
0.5	60.678	19.661	39
1	41.567	29.217	58
1.5	38.424	30.788	62
2	33.246	33.377	67
3	30.880	34.560	69
3.5	30.865	34.567	69

Effect of Initial Arsenate Solution Concentration (AC)

t	time
Co	Initial Concentration
q	As Uptake
%	Percentage of As Uptake

Volume= 0.5 L
AC= 1g
Co = 40 mg/L

Co of 5ppm of AsNaO2			
t	Co	q	%
0	5.000	0.000	0
0.5	1.786	1.607	64
1	0.667	2.167	87
1.5	0.438	2.281	91
2	0.321	2.340	94
3	0.204	2.398	96
3.5	0.191	2.404	96

Co of 20 ppm of AsNaO2			
t	Co	q	%
0	20.000	0.000	0
0.5	10.579	4.711	47
1	5.045	7.477	75
1.5	3.158	8.421	84
2	2.046	8.977	90
3	1.489	9.255	93
3.5	1.075	9.462	95

Co of 40 ppmof AsNaO2			
t	Co	q	%
0	40.000	0.000	0
0.5	25.786	7.107	36
1	10.667	14.667	73
1.5	6.418	16.791	84
2	5.351	17.325	87
3	4.114	17.943	90
3.5	4.104	17.948	90

Co of 100 ppm of AsNaO2			
t	Co	q	%
0	100.000	0.000	0
0.5	60.678	19.661	39
1	39.567	30.217	60
1.5	28.424	35.788	72
2	23.246	38.377	77
3	20.880	39.560	79
3.5	19.865	40.067	80

Effect of pH (CC)

t time
Co Initial Concentration
q As Uptake
% Percentage of As Uptake

Volume= 0.5 L
AC= 1g
Co = 40 mg/L

pH 2			
t	Co	q	%
0	40	0.000	0
0.5	35.7863	2.107	11
1	30.6665	4.667	23
1.5	26.4179	6.791	34
2	20.3507	9.825	49
3	18.1139	10.943	55
3.5	18.1039	10.948	55

pH 5			
t	Co	q	%
0	40	0.000	0
0.5	15.7863	12.107	61
1	10.6665	14.667	73
1.5	5.4179	17.291	86
2	3.3507	18.325	92
3	2.1139	18.943	95
3.5	2.1039	18.948	95

pH 7			
t	Co	q	%
0	40	0.000	0
0.5	15.7863	12.107	61
1	10.6665	14.667	73
1.5	6.4179	16.791	84
2	5.3507	17.325	87
3	4.1139	17.943	90
3.5	4.1039	17.948	90

pH 8			
t	Co	q	%
0	40	0.000	0
0.5	36.7863	1.607	8
1	32.6665	3.667	18
1.5	29.4179	5.291	26
2	25.3507	7.325	37
3	24.1139	7.943	40
3.5	24.1039	7.948	40

Effect of amount of adsorbent (CC)

t time
Co Initial Concentration
q As Uptake
% Percentage of As Uptake

Volume= 0.5 L
AC= 1g
Co = 40 mg/L

0.2 g			
t	Co	q	%
0	40.000	0.000	0
0.5	36.786	1.607	8
1	36.667	1.667	8
1.5	35.418	2.291	11
2	35.351	2.325	12
3	34.114	2.943	15
3.5	34.003	2.998	15

0.8 g			
t	Co	q	%
0	40.000	0.000	0
0.5	29.786	5.107	26
1	28.667	5.667	28
1.5	27.418	6.291	31
2	26.351	6.825	34
3	25.114	7.443	37
3.5	23.452	8.274	41

1.0 g			
t	Co	q	%
0	40.000	0.000	0
0.5	15.232	12.384	62
1	8.245	15.878	79
1.5	6.238	16.881	84
2	5.147	17.427	87
3	4.044	17.978	90
3.5	2.044	18.978	95

2.0 g			
t	Co	q	%
0	40.000	0.000	0
0.5	28.678	5.661	28
1	20.567	9.717	49
1.5	18.424	10.788	54
2	16.946	11.527	58
3	16.880	11.560	58
3.5	16.635	11.683	58